



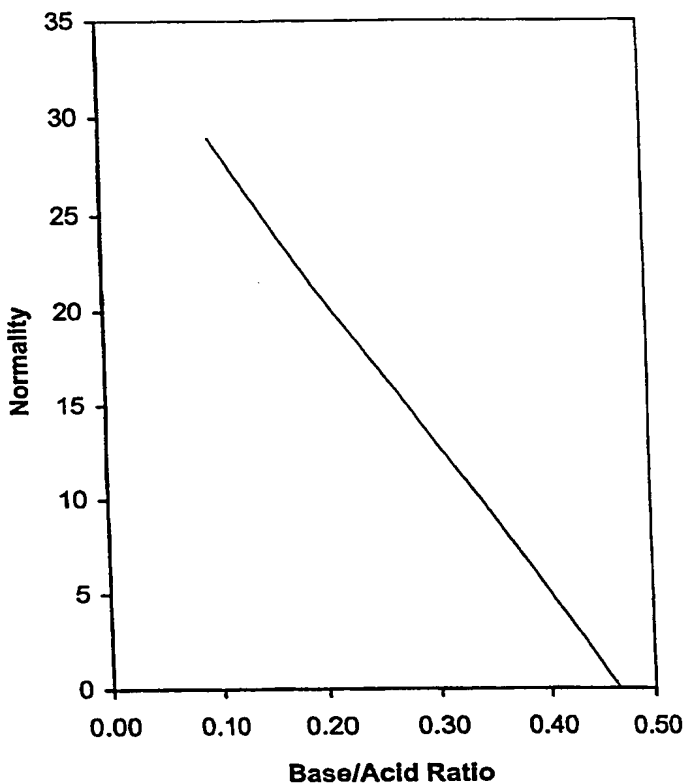
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(71) Applicant (for all designated States except US): MORN- INGSTAR DIAGNOSTICS [US/US]; Suite 130, 1376 Lead Hill Boulevard, Roseville, CA 95661 (US).			
(72) Inventors; and (75) Inventors/Applicants (for US only): KEMP, Maurice, Clarence [US/US]; 4401 Woodthrush Drive, El Dorado Hills, CA 95762 (US). LALUM, Robert, Blaine [US/US]; 6949 Green- back Lane #B, Citrus Heights, CA 95621 (US). XIE, Zhong, Wei [US/US]; 1141 Elderberry Circle, Folsom, CA 95630 (US). CUNHA, Michael, Anthony [US/US]; 355 Shadow Ridge Drive #3, Roseville, CA 95678 (US). CARPENTER, Robert, H. [US/US]; 1303 Pecan Street, Bastrop, TX 78602 (US). ZHANG, Shu [US/US]; 401 Rocky Point Drive #5, Roseville, CA 95678 (US). YAO, Yu [US/US]; 401 Rocky Point Drive #5, Roseville, CA 95678 (US). LEWIS, David, E. [US/US]; 816 3rd Avenue, Eau Claire, WI 54703 (US).		Published Without international search report and to be republished upon receipt of that report.	

(54) Title: ADDUCT HAVING AN ACIDIC SOLUTION OF SPARINGLY-SOLUBLE GROUP IIA COMPLEXES

## (57) Abstract

An adduct that has an acidic solution of sparingly-soluble Group IIA complexes ("AGIIS") and at least one additive. The AGIIS can be prepared by mixing a mineral acid (such as sulfuric acid), and a Group IIA hydroxide (such as calcium hydroxide) or a Group IIA salt of a dibasic acid (such as calcium sulfate), or a mixture of the two Group IIA compounds, followed by removing the solid formed. The additives can be an alcohol, an organic acid or a surface active agent. The composition has various uses, including cleaning, food production, decontamination, bioremediation, agricultural application, medical application, and detoxification of substances.



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ADDUCT HAVING AN ACIDIC SOLUTION OF SPARINGLY-SOLUBLE  
GROUP IIA COMPLEXES

BACKGROUND

This application is a continuation-in-part of an application filed February 19, 1999, Serial No. 09/253,241, the entire content of which is incorporated by reference.

5 This invention relates to an adduct, a composition having an acidic solution of sparingly-soluble group IIA complexes ("AGIIS") and at least one additive, to its preparation, and to its uses.

In the late 80's and early 90's, researchers in Japan  
10 developed strong ionized water ("SIW") as disinfectants. The SIW was established as water with pH 2.7 or less, having an oxidation-reduction potential of 1,000 mv or more, and chlorine concentration of 0.8 ppm or more. The SIW is prepared by electrolysis of water.

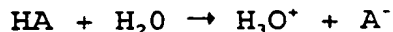
15 Electrolysis of tap water has also been used to produce "strong acid water" and "strong alkali water" both of which were claimed to have antiseptic properties.

U.S. Patent Number 5,830,838 to Wurzbarger, et al. describes a solution for cleaning metal surfaces. The  
20 solution is prepared by mixing calcium hydroxide and potassium hydroxide with equivalent of sulfuric acid in water then passing the solution through a 10 micron filter. The resulting concentrate can be diluted depending on the degree of surface oxidation of the metal to be treated.

U.S. Patent Number 5,895,782 to Overton, et al. describes a solution for cleaning metal surfaces particularly non-ferrous alloys such as copper, brass and high strength aluminum alloys. The solution is prepared by  
5 mixing  $\text{Ca}(\text{OH})_2$  and KOH with equivalent sulfuric acid in water then passing the solution through a 10 micron filter. The resulting concentrate can be used full strength or diluted depending on the degree of surface oxidation of the metal to be treated.

10 International Publication WO 94/09798 describes a pharmaceutical composition for treatment of disease, injury and other disorders. The pharmaceutical composition comprises a complex of a calcium-containing component and a sulfate-containing component in a pharmaceutically  
15 acceptable carrier. The reference teaches the isolation from natural materials, such as peat, the inorganic compositions. The inorganic preparations comprise an alkaline, aqueous or organic, or mixture thereof, extract of peat. Peat is extracted with aqueous solutions, organic  
20 solutions or water-miscible organic solvents at temperature from below room temperature up to the boiling point of the solvents. The preferred extracting solvents are those having a pH of at least 9. Biologically active constituents of fractionated peat preparations were  
25 identified as  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (gypsum),  $\text{CaSO}_4 \cdot \text{K}_2\text{SO}_4 \cdot \text{H}_2\text{O}$  (syngenite, also referred to as the double salt of gypsum) and  $\text{K}_3\text{Na}(\text{SO}_4)_2$  (apthitalite) by X-ray powder diffraction analysis. The reference also describes the synthesis of syngenite.

Chemists describe and measure the ability of a  
30 substance to donate protons  $[\text{H}^+]$  to a chemical reaction as the pka of that substance where



Although a hydronium ion is usually represented by  $\text{H}^+$  or  $\text{H}_3\text{O}^+$ , but its true formula is not certain. The aggregate  
5 could be  $\text{H}_5\text{O}_2^+$ ,  $\text{H}_7\text{O}_3^+$ , or even  $\text{H}_9\text{O}_4^+$ .

The ability to produce large quantities of positively charged water would be extremely important in a large number of applications where water is the medium of the reaction. Positively charged water has the ability to  
10 donate protons [ $\text{H}^+$ ]. The donation of a proton is usually an intermediate step in any acid hydrolysis reaction. Acids are usually the chemical reagent used to donate protons in an aqueous solution. If the water could be the source of the [ $\text{H}^+$ ], then there would be fewer unwanted by-products  
15 (toxins) from the reactions and there would be less hazard associated with these products use.

A strong acid is used to neutralize and remove the lime , or quicklime, from the brick and mortar. A strong acid, such as hydrochloric acid, also known as muriatic  
20 acid, is also used to clean hard water spots on shower stalls, windows, glass, toilets, urinals, mirrors and other surfaces. Hydrochloric acid is used to de-scale water towers and heat exchangers and to adjust the pH of the waste water effluent.

25 A full strength mineral acid, such as hydrochloric acid, is extremely corrosive to many substances, including metals. In addition, hydrochloric acid at a low pH of 0.5 or so will burn a human skin in seconds. The acid is also very harmful in that it emits fumes irritating to mucous  
30 membrane. If left near other chemicals, like bleach, hydrochloric acid will interact with them, even through a typical plastic bottle.

It is thus desirable to be able to have source of "acidity," or  $H_3O^+$ , without these unwanted disadvantages and be able to reduce environmental and safety hazards associated with acid hydrolysis. Preferably, this source of

5 "acidity" should be able to prevent re-contamination following decontamination, not induce bacterial resistance, not alter the taste, color or smell of treated foodstuffs, not create any odor, effective in water in a wide range of temperatures, relatively free of danger when overdosed, can

10 be neutralized after use, not carcinogenic or mutagenic, non-toxic, almost harmless in water and the environment, environmentally friendly, and can be stored for a long period of time without decomposition or turning into hazardous compound.

15 The control of microbial growth is necessary in many practical situations, and significant advances in agriculture, medicine and food science have been made through study of this area of microbiology. "Control of growth" means to prevent growth of microorganisms. This

20 control is effected in one of two basic ways: (1) By killing microorganisms; or (2) by inhibiting the growth of microorganisms. Control of growth usually involves the use of physical or chemical agents which either kill or prevent the growth of microorganisms. Agents which kill cells are

25 called "cidal" agents; agents which inhibit the growth of cells, but without killing them, are referred to as "static" agents. Thus the term "bactericidal" refers to killing bacteria and "bacteriostatic" refers to inhibiting the growth of bacterial cells. A "bactericide" kills

30 bacteria, a "fungicide" kills fungi. "Sterilization" is the complete destruction or elimination of all viable organisms in or on an object being sterilized. The object

is either sterile or not, there are no degrees of sterilization. Sterilization procedures involve the use of heat, radiation or chemicals, or physical removal of microorganisms.

5        Microorganisms tend to colonize and replicate on different surfaces resulting in adherent heterogenous microbial accumulations termed "biofilms." Biofilms may form on surfaces of food substances, feed substances, and instrumentations. The microorganisms in the biofilms may  
10 include bacteria, fungi, viruses, and protozoans. Since food safety is a national priority, any product that can help by solving a multitude of problems associated with food production is desirable. Removal and control of biofilms which harbor dangerous microbial contamination is  
15 a sanitation goal that needs to be achieved. It is also desirable to be able to safely decontaminate water and nutriment by lowering pH to levels where contaminants would react and organisms cannot live.

As used herein, the term "nutriment" means something  
20 that nourishes, heals, or promotes growth and repair the natural wastage of organic life. Thus, food for a human and feed for an animal are all examples of nutriment. Other examples of nutriment include beverages, food additive, feed additive, beverage additive, food supplement, feed  
25 supplement, beverage supplement, seasoning, spices, flavoring agent, stuffing, food dressing, pharmaceutical, biological product, and others. The nutriment can be of plant origin, animal origin, or synthetic. C u r r e n t sanitizing, disinfectants and pesticides products on the  
30 market for these uses contain residues of chlorine, ammonia, organic iodine, metal salts and other deleterious residues. It is desirable to have a way that would preclude

these residues by promoting acid hydrolysis without the presence of deleterious chemicals. Additionally, this method should generate few hazardous volatile gases. Importantly, it is highly desirable to have a composition  
5 that can control and the growth of, and kill, microorganisms and, at the same time, destroy the products, such as toxins, generated by, or associated with, the microorganisms.

10



### SUMMARY

One embodiment of the present invention involves an adduct which is a composition containing a acidic solution  
5 of sparingly-soluble Group IIA complex ("AGIIS") and an additive. In a way, the additive enhances, and is synergistic to, the effectiveness of the AGIIS. Other aspects of the present invention pertain to different methods to promote the safe, clean, and environmentally  
10 sensitive ways of chemical production, pharmaceutical production, cleaning, food production, decontamination, bioremediation, agricultural application, medical application, and detoxification of a wide variety of substances.

15

### DESCRIPTION OF THE FIGURE

Figure 1 shows the relation of the desired final acid normality of AGIIS and the mole ratio of calcium hydroxide to sulfuric acid, given in moles of calcium hydroxide per  
20 mole of sulfuric acid.

### DETAILED DESCRIPTION

One aspect of the present invention pertains to an adduct containing an acidic, or low pH, solution of  
25 sparingly-soluble Group IIA complexes ("AGIIS") and an additive. The solution may have a suspension of very fine particles and the term "low pH" means the pH is below 7, in the acidic region. The AGIIS of the present invention with a certain acid normality does not have the same dehydrating  
30 behavior as a saturated calcium sulfate in sulfuric acid having the same normality. In other words, the AGIIS of the present invention with a certain acid normality does

not char sucrose as readily as does a saturated solution of calcium sulfate in sulfuric acid having the same normality. Further, the AGIIS is non-volatile at room temperature. It is less corrosive to a human skin than sulfuric acid  
5 saturated with calcium sulfate having the same acid normality. Not intending to be bound by the theory, it is believed that one embodiment of AGIIS comprises near-saturated, saturated, or super-saturated calcium, sulfate anions or variations thereof, and/or complex ions  
10 containing calcium, sulfates, and/or variations thereof.

The term "complex," as used herein, denotes a composition wherein individual constituents are associated. "Associated" means constituents are bound to one another either covalently or non-covalently, the latter as a result  
15 of hydrogen bonding or other inter-molecular forces. The constituents may be present in ionic, non-ionic, hydrated or other forms.

The acidic solution of sparingly-soluble Group IIA-complex salt ("AGIIS") can be prepared in several ways.  
20 Some of the methods involve the use of Group IA hydroxide but some of syntheses are devoid of the use of any added Group IA hydroxide, although it is possible that a small amount of Group IA metal may be present as "impurities." The preferred way of manufacturing AGIIS is not to add  
25 Group IA hydroxide to the mixture. As the phrase implies, AGIIS is highly acidic, ionic, with a pH of below about 2.

Wurzbarger, et al. in U.S. Patent 5,830,838 describes an acidic solution prepared by the "calcium-hydroxide/potassium-hydroxide method." The solution is  
30 produced by first adding two moles of concentrated sulfuric acid (93%) to 2 liters of de-ionized water. Separately, an aqueous solution of base is prepared by adding one mole of

calcium hydroxide (hydrated lime) and two moles of potassium hydroxide to 20 liters of de-ionized water with stirring. The acid solution is then mixed with the base solution. The mixture is then filtered through a 10 micron filter to remove particles of calcium sulfate or potassium sulfate of eleven microns or larger. The resulting concentrate can be used full strength or diluted with water depending on the metal surfaces to be treated. Sodium hydroxide may be used in place of potassium hydroxide. Hydrated calcium oxide may be used in place of calcium hydroxide. Another source of the base is calcium metal. In either case and as one embodiment of this application, the resultant solution is a highly acidic solution. This highly acidic solution can be diluted with water to adjust its pH to a desired higher value, i.e. less acidic.

Another way of preparing the acidic solution is by the "calcium-metal method" which involves reacting concentrated sulfuric acid with calcium metal followed by filtration. One mole of concentrated sulfuric acid was diluted with 40 moles of de-ionized water. Then, one mole of calcium metal turnings was slowly added with stirring into the solution of sulfuric acid. The stirring was continued until essentially all metal had dissolved. The resultant mixture was allowed to settle for about 5 to 6 hours before the supernatant was filtered through a 10 micron filter. The concentrate thus obtained had a pH value of about 0.5. This concentrate of hydronium ions was then diluted with de-ionized water to the desired pH value, such as pH of about 1 or about 1.8.

Then, there is the "calcium-hydride method" which involves reacting concentrated sulfuric acid and calcium

hydride in water. One mole of concentrated sulfuric acid was diluted with 40 moles of de-ionized water. With agitation, 1 mole of calcium hydride was slowly added to the solution of sulfuric acid. The agitation was  
5 continued until the calcium hydride has essentially all dissolved. After the dissolution, the mixture was then allowed to settle for about 5 to 6 hours, at that time the supernatant was filtered through a 10 micron filter. The concentrate thus obtained had a pH value of about 0.1 to  
10 about 0.2, and can be further diluted.

One product from the "calcium-metal method" or "calcium-hydride method" having a pH of from -0.2 to -0.3, and from 1.4 to 1.5 acid normality gave the following analyses: Ca, 763 ppm; SO<sub>4</sub>, 84633 ppm; Na, 4.76 ppm; K,  
15 3.33 ppm; and Mg, 35.7 ppm.

The "calcium-metal method" and the "calcium-hydride method" have certain drawbacks. In each of these methods, thermal control is very difficult to achieve because of the large amount of heat generated when concentrated sulfuric  
20 acid is reacted with either calcium metal or calcium hydride. The difficulties in thermal control of the reactions cause the reactions to be difficult to reproduce and hard to control.

The preferred method of preparing AGIIS involves  
25 mixing a mineral acid with a Group IIA hydroxide, or with a Group IIA salt of a dibasic acid, or with a mixture of the two Group IIA materials. In the mixing, a salt of Group IIA is also formed. Preferably, the starting Group IIA material or materials selected will give rise to, and  
30 form, the Group IIA salt or salts that are sparingly soluble in water. The preferred mineral acid is sulfuric acid, the preferred Group IIA hydroxide is calcium

hydroxide, and the prefer Group IIA salt of a dibasic acid is calcium sulfate. Other examples of Group IIA salt include calcium oxide, calcium carbonate, and "calcium bicarbonate."

5        Thus, for example, AGIIS can be prepared by mixing or blending starting materials given in one of the following scheme with good reproducibility:

- (1)  $\text{H}_2\text{SO}_4$  and  $\text{Ca}(\text{OH})_2$ ;
- (2)  $\text{H}_2\text{SO}_4$ ,  $\text{Ca}(\text{OH})_2$ , and  $\text{CaCO}_3$ ;
- 10    (3)  $\text{H}_2\text{SO}_4$ ,  $\text{Ca}(\text{OH})_2$ ,  $\text{CaCO}_3$ , and  $\text{CO}_2$  (gas);
- (4)  $\text{H}_2\text{SO}_4$  and  $\text{CaCO}_3$ ;
- (5)  $\text{H}_2\text{SO}_4$ ,  $\text{CaCO}_3$ , and  $\text{Ca}(\text{OH})_2$ ;
- (6)  $\text{H}_2\text{SO}_4$ ,  $\text{CaCO}_3$ , and  $\text{CO}_2$  (gas);
- (7)  $\text{H}_2\text{SO}_4$  and  $\text{CaSO}_4$ ;
- 15    (8)  $\text{H}_2\text{SO}_4$ ,  $\text{Ca}(\text{OH})_2$ , and  $\text{CaSO}_4$ ;
- (9)  $\text{H}_2\text{SO}_4$ ,  $\text{CaSO}_4$ , and  $\text{CaCO}_3$ ;
- (10)  $\text{H}_2\text{SO}_4$ ,  $\text{CaSO}_4$ ,  $\text{CaCO}_3$ , and  $\text{Ca}(\text{OH})_2$ ;
- (11)  $\text{H}_2\text{SO}_4$ ,  $\text{CaSO}_4$ ,  $\text{CaCO}_3$ , and  $\text{CO}_2$  (gas); and
- (12)  $\text{H}_2\text{SO}_4$ ,  $\text{CaSO}_4$ ,  $\text{CaCO}_3$ ,  $\text{CO}_2$  (gas), and  $\text{Ca}(\text{OH})_2$ .

20

Thus, preferably, AGIIS is prepared by mixing calcium hydroxide with concentrated sulfuric acid, with or without an optional Group IIA salt of a dibasic acid (such as calcium sulfate) added to the sulfuric acid. The optional  
25 calcium sulfate can be added to the concentrated sulfuric acid prior to the introduction of calcium hydroxide into the blending mixture. The addition of calcium sulfate to the concentrated sulfuric acid appears to reduce the amount of calcium hydroxide needed for the preparation of AGIIS.  
30 Other optional reactants include calcium carbonate and gaseous carbon dioxide being bubbled into the mixture.

Regardless of the use of any optional reactants, it was found that the use of calcium hydroxide is desirable.

One preferred method of preparing AGIIS can be described briefly as: Concentrated sulfuric acid is added  
5 to chilled water ( $8^{\circ}$  -  $12^{\circ}\text{C}$ ) in the reaction vessel, then, with stirring, calcium sulfate is added to the acid in chilled water to give a mixture. Temperature control is paramount to this process. To this stirring mixture is then added a slurry of calcium hydroxide in water. The  
10 solid formed from the mixture is then removed. This method involves the use of sulfuric acid, calcium sulfate, and calcium hydroxide, and it has several unexpected advantages. Firstly, this reaction is not violent and is not exceedingly exothermic. Besides being easy to control  
15 and easy to reproduce, this reaction uses ingredients each of which has been reviewed by the U.S. Food and Drug Administration ("U.S. FDA") and determined to be "generally recognized as safe" ("GRAS"). As such, each of these ingredients can be added directly to food, subject,  
20 of course, to certain limitations. Under proper concentration, each of these ingredients can be used as processing aids and in food contact applications. Their use is limited only by product suitability and Good Manufacturing Practices ("GMP"). The AGIIS so prepared is  
25 thus safe for animal consumption, safe for processing aids, and safe in food contact applications. Further, the AGIIS reduces biological contaminants in not only inhibiting the growth of, and killing, microorganisms but also destroying the toxins formed and generated by the microorganisms. The  
30 AGIIS formed can also preserve, or extend the shelf-life of, consumable products, be they plant, animal, pharmaceutical, or biological products. It also preserves

or improves the organoleptic quality of a beverage, a plant product or an animal product. It also possesses certain healing and therapeutic properties.

The sulfuric acid used is usually 95-98% FCC Grade  
5 (about 35-37 N). The amount of concentrated sulfuric acid can range from about 0.05 M to about 18 M (about 0.1 N to about 36 N), preferably from about 1 M to about 5 M. It is application specific. The term "M" used denotes molar or moles per liter.

10 Normally, a slurry of finely ground calcium hydroxide suspended in water (about 50% of W/V) is the preferred way of introducing the calcium hydroxide, in increments, into the a stirring solution of sulfuric acid, with or without the presence of calcium sulfate. Ordinarily, the reaction  
15 is carried out below 40°C, preferably below room temperature, and more preferably below 10°C. The time to add calcium hydroxide can range from about 1 hour to about 4 hours. The agitation speed can vary from about 600 to about 700 rpm or higher. After the mixing, the mixture is  
20 filtered through a 5 micron filter. The filtrate is then allowed to sit overnight and the fine sediment is removed by decantation.

The calcium hydroxide used is usually FCC Grade of about 98% purity. For every mole of concentrated acid,  
25 such as sulfuric acid, the amount, in mole, of calcium hydroxide used is application specific and ranges from about 0.1 to about 1.

The optional calcium carbonate is normally FCC Grade having a purity of about 98%. When used with calcium  
30 hydroxide as described above, for every mole of concentrated acid, such as sulfuric acid, the amount, in

mole, of calcium carbonate ranges from about 0.001 to about 0.2, depending on the amount of calcium hydroxide used.

The optional carbon dioxide is usually bubbled into the slurry containing calcium hydroxide at a speed of from  
5 about 1 to about 3 pounds pressure. The carbon dioxide is bubbled into the slurry for a period of from about 1 to about 3 hours. The slurry is then added to the reaction vessel containing the concentrated sulfuric acid.

Another optional ingredient is calcium sulfate, a  
10 Group IIA salt of a dibasic acid. Normally, dihydrated calcium sulfate is used. As used in this application, the phrase "calcium sulfate," or the formula " $\text{CaSO}_4$ ," means either anhydrous or hydrated calcium sulfate. The purity of calcium sulfate (dihydrate) used is usually 95-98% FCC  
15 Grade. The amount of calcium sulfate, in moles per liter of concentrated sulfuric acid ranges from about 0.005 to about 0.15, preferably from about 0.007 to about 0.07, and more preferably from about 0.007 to about 0.04. It is application specific.

20 From experimental data, a slope was generated showing the ratio of calcium hydroxide to concentrated sulfuric needed for a desired final acid normality of AGIIS. See, Figure 1.

The slope in Figure 1 was created from two pairs of  
25 data points found by titrating a given amount of acid to a desired final acid normality. The accuracies were determined chemically. The final acid normality of the finished product ranges from about 1.2 to about 29. To produce one liter of 1.2 N AGIIS, it was found that for  
30 every mole of concentrated sulfuric acid, 0.45 moles of  $\text{Ca}(\text{OH})_2$  was required. To produce one liter of 27 N AGIIS, it was found that for every mole of concentrated sulfuric



acid, 0.12 moles of  $\text{Ca}(\text{OH})_2$  was required. The data were then plotted onto a graph where the Y-axis represents final acid normality and the X-axis represents moles of  $\text{Ca}(\text{OH})_2$ /1 mole of concentrated sulfuric acid, where  $X_1 = 0.45$ ,  $X_2 =$   
5 0.12,  $Y_1 = 1.2$ , and  $Y_2 = 27$ . The slope of the line was found by using the equation  $(Y_1 - Y_2)/(X_1 - X_2)$ , which was -78.18. The line can be represented by the equation  $Y = mX + b$ , where  $mX$  is the slope, and  $b$  is the Y intercept. The highest acid normality was 36.65, thus the equation is:  
10  $Y = -78.18X + 36.65$

This slope is useful for the preparation of an AGIIS solution having a desired final acid normality.

Broadly, the method of preparing AGIIS having a  
15 desired final acid normality involves the steps given below. The calculations are based on a 1 liter of final volume of AGIIS, the amounts of acid (concentrated sulfuric acid) and base (calcium hydroxide) are in moles, the ratio of base to acid is the number of moles of base (calcium  
20 hydroxide) for every mole of acid (concentrated sulfuric acid). The steps are:

(a) Determining the amount of mineral acid (such as concentrated sulfuric acid), in moles, needed to produce AGIIS having the desired final acid normality ("N") by  
25 using a relationship given by the following equation:

$$E_1 = (N/2) + (N/2 + B)$$

in which  $E_1$  is the amount of acid, in moles, required before correcting for purity, or purity adjustment;  $N$  is the desired final acid normality; and  $B$  is  
30 the mole ratio of the Group IIA hydroxide to the mineral acid needed to obtain the AGTIIS having  $N$ , and  $B$  is derived from a pre-plotted curve depicting the relationship of the

mineral acid and the Group IIA hydroxide for a desired final N;

- (b) making purity adjustment for the mineral acid used. The correction for the purity of the acid used is accomplished by the equation:

$$E_2 = E_1/C$$

in which  $E_2$  is the amount of acid, in moles, required after correcting for purity of the acid used, or purity adjustment;  $E_1$  is as defined above; and C is purity adjustment factor for the acid used. For concentrated sulfuric acid, the average acid strength is about 96.5%, and thus C is 0.965;

- (c) determining the amount of water, in ml, that has to be added to the acid whose acid solution will then, after the reaction, give the desired final acid normality N. The relationship is as follows:

$$G = J - E_2 - I$$

in which G is the amount of water required to be added to the mineral acid solution to get the desired final acid normality; J is the final volume of the aqueous mineral acid solution; I is the volume amount of Group IIA hydroxide needed (see, below); and  $E_2$  is as defined above;

- (d) adding G to  $E_2$  to give the final aqueous solution of the mineral acid, in which both G and  $E_2$  are as defined above;

- (e) determining the amount of base, (such as calcium hydroxide), in moles, needed for the reaction to produce AGIIS having the desired final acid normality N. For example, from the straight line in Figure 1, the mole ratio of  $\text{Ca}(\text{OH})_2$  to concentrated  $\text{H}_2\text{SO}_4$  to achieve a certain final acid normality can be determined.

the amount of the base, in moles, needed is:

$$F_1 = N/2 \times B$$

in which  $F_1$  is the amount of base, in moles, needed; and N and B are as defined above;

(f) the correction for the purity of the base used is  
5 accomplished by the equation:

$$F_2 = F_1/D$$

in which  $F_2$  is the amount of base, in moles, required after correcting for purity of the base used, or purity adjustment; and D is purity adjustment factor for  
10 the base used.

The average purity of sodium hydroxide is about 98%, and, thus, D, in this case, is 0.98;

(g) determining the amount of water, in ml, needed to make the slurry of base. The relationship is as follows:

15 
$$H = F_2 \times 1.5$$

in which H is the volume of water, in ml, needed to make the slurry of base which, in turn, will give AGIIS with the desired final acid normality N.  $F_2$  is as defined above. The H given is an approximation and should be  
20 adjusted to a desired final weight volume. Thus, for example, 50 g of base should be adjusted to a final volume of 100 ml because the slurry used is a 50 : 50 mixture of solid and water;

(h) determining the volume, in ml, of the base slurry  
25 or solution to be added to the acid solution to give AGIIS with the desired final acid normality N. The relationship can be expressed as:

$$I = F_2 \times 2$$

in which I is the volume, in ml, of the slurry or  
30 solution of base to be added to the acid solution; and  $F_2$  is as defined above;

(i) adding H to F<sub>2</sub> to give the final aqueous slurry or solution of the base, in which both H and F<sub>2</sub> are as defined above;

(j) adding the final aqueous solution or slurry or the base of (i) to the final aqueous solution of mineral acid of (d);

(k) allowing the final aqueous solution or slurry of the base and the final aqueous solution of mineral acid (j) to react; and

(l) removing solid formed from the reaction of (k).

In the event that CaSO<sub>4</sub> is used for the reaction by adding it to the solution of concentrated H<sub>2</sub>SO<sub>4</sub>, the amount of CaSO<sub>4</sub>, in grams per liter of solution based on final volume, has the following relationship:

	<u>Final AGIIS Acid Normality N</u>	<u>Amount of CaSO<sub>4</sub> in g/l</u>
	1 - 5	5
	6-10	4
20	11-15	3
	16-20	2
	21-36	1

The AGIIS obtained could have an acid normality range of from about 0.05 to about 31; the pH of lower than 0; boiling point of from about 100 to about 106°C; freezing point of from about -8°C to about 0°C.

AGIIS obtained from using the reaction of H<sub>2</sub>SO<sub>4</sub>/Ca(OH)<sub>2</sub>/CaSO<sub>4</sub> had the following analyses (average):

AGIIS With Final Acid Normality of 1.2 N, pH of  
-0.08

H<sub>3</sub>O<sup>+</sup>, 2.22%; Ca, 602 ppm; SO<sub>4</sub>, 73560 ppm; K, 1.36  
ppb; impurities of 19.68 ppm, and neither Na nor Mg was  
5 detected.

AGIIS With Final Acid Normality of about 29 N, pH of  
about -1.46

H<sub>3</sub>O<sup>+</sup>, 30.68%; Ca, 52.9 ppm; SO<sub>4</sub>, 7356000 ppm; K,  
10 38.02 ppb; and neither Na nor Mg was detected.

Besides concentrated sulfuric acid, other polyprotic  
acids, such as phosphoric acid, phosphorous acid, chloric  
acid, iodic acid, or others can be used.

15 Likewise, aqueous solutions of other alkalines or  
bases, such as Group IA hydroxide solution or slurry and  
Group IIA hydroxide solution or slurry can be used. Groups  
IA and IIA refer to the two Groups in the periodical table.  
The use of Group IIA hydroxide is preferred. Preferably,  
20 the salts formed from using Group IIA hydroxides in the  
reaction are sparingly-soluble in water. It is also  
preferable to use only Group IIA hydroxide as the base  
without the addition of Group IA hydroxide.

After the reaction, the resultant concentrated acidic  
25 solution with a relatively low pH value, typically below pH  
1, can then be diluted with de-ionized water to the desired  
pH value, such as pH of about 1 or about 1.8.

However, it is sometimes desirable not to prepare a  
very concentrated AGIIS solution and then dilute it  
30 serially to obtain the solution having the desired final  
acid normality. It is often desirable to prepare a  
solution of AGIIS having a desired final pre-determined

acid normality according to the method described in this application so that not much dilution of the product is required before use.

As discussed above, AGIIS has relatively less  
5 dehydrating properties (such as charring sucrose) as compared to the saturated solution of  $\text{CaSO}_4$  in the same concentration of  $\text{H}_2\text{SO}_4$ . Further, the stability and non-corrosive nature of the AGIIS of the present invention can be illustrated by the fact that a person can put his or her  
10 hand into this solution with a pH of less than 0.5 and, yet, his or her hand suffers no irritation, and no injury. If, on the other hand, one places his or her hand into a solution of sulfuric acid of pH of less than 0.5, an irritation would occur within a relatively short span of  
15 time. A solution of 28 N of sulfuric acid saturated with calcium sulfate will cause chemical burn to a human skin after a few seconds of contact. In contrast, AGIIS solution of the same normality would not cause chemical  
20 The AGIIS of the present invention does not seem to be corrosive when being brought in contact with the environmental protective covering of plants (cuticle) and animals (skin). AGIIS is non-volatile at room temperature. Even as concentrated as 29 N, the AGIIS has no odor, does  
25 not give off fumes in the air, and is not irritating to a human nose when one smells this concentrated solution.

The "additive" of the present invention appears to enhance, and also appears to be synergistic to, the effectiveness of the AGIIS. Examples of the additive  
30 include alcohol, organic acid, periodic acid, and surfactant. The amount of additive added to the AGIIS varies depending on the desired final weight percent of the

additive in the final adduct composition. The weight percent of additive needed for the adduct composition of the present invention can vary from about 0.01 to about 99.99, based on the total weight of the final adduct composition. In one aspect, if the additive is to be added to the concentrated AGIIS with a very low pH value, then the amount of the additive added has to be adjusted in anticipation of further dilution with water to raise the pH value of the final adduct composition. The alcohol additive preferred for the present invention includes methanol, ethanol, propanol, i-propanol, and other lower alkyl alcohols.

Organic acid additive of the present invention includes carboxylic acid. A carboxylic acid is an organic compound containing the -COOH group, i.e., a carbonyl attached to a hydroxyl group. Preferred organic acids for the present invention include lactic acid, acetic acid, propionic acid, oxalic acid, and per-acetic acid.

A surfactant for the present invention is a surface-active agent. It is usually an organic compound consisting of two parts: One, a hydrophobic portion, usually including a long hydrocarbon chain; and two, a hydrophilic portion which renders the compound sufficiently soluble or dispersible in water or another polar solvent. Surfactants are usually classified into: (1) an-ionic, where the hydrophilic moiety of the molecule carries a negative charge; (2) cat-ionic, where this moiety of the molecule carries a positive charge; and (3) non-ionic, which do not dissociate, but commonly derive their hydrophilic moiety from polyhydroxy or polyethoxy structures. Other surfactants include ampholytic and zwitterionic

surfactants. A preferred surfactant for the present invention includes polysorbates (Tween 80).

Unless otherwise defined, the amount of each ingredient or component of the present invention is based on the weight percent of the final composition, usually the concentrate before further dilution to achieve the desired pH of about 1.8. The AGIIS having a pH of about 1.8 is usually further diluted with water before applying to an animal product or a plant product.

One way of preparing a concentrate of the AGIIS having an ethanol additive and a lactic acid additive is by mixing with stirring at ambient temperature 634 mL of 200 proof FCC ethanol (16.5 weight %); 75 mL. of 85% lactic acid (1.9 weight %); 1536 mL of a solution of AGIIS having a pH of about 0.2 - 0.4 (40 weight %); and 1595 mL of de-ionized water (41.5 weight %). The resultant concentrate of AGIIS with two additives showed a pH of about 1.65 -1.8.

One way of preparing a concentrate of the AGIIS having ethanol, lactic, and Tween 80 additives is by mixing with stirring at ambient temperature 634 mL of 200 proof FCC ethanol (16.5 weight %); 75 mL. of 85% lactic acid (1.9 weight %); 1920 mL of a solution of AGIIS having a pH of about 0.2 - 0.4 (50 weight %); 255 mL of Tween 80 (6.6 weight %); and 957.6 mL of de-ionized water (25 weight %). The resultant concentrate of AGIIS with three additives showed a pH of about 1.45 - 1.7.

A "biological contaminant" is defined as a biological organism, or the product of biological organism, such as toxin, all of which contaminate the environment and useful products. This biological contaminant results in making the environment or product hazardous.



Biological contaminants, such as bacteria, fungi, mold, mildew, spores, and viruses have potentially reactive substances in their cell wall/membranes; however, they hide in cells (viruses and some bacteria) and/or secrete biofilms  
5 (most bacteria, fungi, mold and mildew) to protect them from the environment.

Bacterial form or elaborate intracellular or extracellular toxins. Toxin is a noxious or poisonous substance that: (1) is an integral part of the bacteria;  
10 (2) is an extracellular product (exotoxin) of the bacteria; or (3) represents a combination of the two situations, formed or elaborated during the metabolism and growth of bacteria. Toxins are, in general, relatively complex antigenic molecules and the chemical compositions are  
15 usually not known. The harmful effects of bacteria come not only from the bacteria themselves, but also from the toxins produced by bacteria. Toxins produced by bacteria are just as, if not more, hazardous to the product than the bacteria themselves. Ordinary disinfectants, such as  
20 quaternary ammonium compounds, will kill bacteria but have no effect on bacterial toxins and endotoxins. In fact, many disinfectants actually contribute to the endotoxins problems by causing their release from the bacteria. The bacterial toxins and endotoxins can cause serious adverse  
25 effects in human and animals. Endotoxins are the major cause of contamination in food products, in the production of pharmaceuticals, medical devices, and other medical products. Thus, while "decontaminating" a product infested with bacteria, it is not enough to simply kill or reduce  
30 the number of bacteria. To get a safe and decontaminated product, the toxins and endotoxins of the bacteria must also be destroyed. Neither killing the microorganism alone

nor destroying the toxins alone is enough in the real world. To be useful, when reducing biological contaminants in a nutriment or in an equipment, the growth of biological organisms must be controlled and reduced, and, at the same  
5 time, the product of biological organisms (such as toxins) must be removed and/or destroyed.

The outer covering, i.e. epidermis, of animals and cuticle of plants resist the growth and/or entry of the above microorganisms into the interior of the complex  
10 organism. One of the microbial growth prevention methods used by plants and animals is the maintenance of a surface pH or secretion of a coating that is not conducive to the attachment and propagation of micro-organisms. After a plant product is harvested or an animal product processed,  
15 these products loose the ability to resist the infestation of micro-organisms. By spraying the composition of the present invention plus defined additives on fruits, vegetables, and whole plants post harvest or washing or packing animal products in the composition, the growth and  
20 propagation of micro-organisms in these products can be reduced. If plant or animal products are packed in the composition an additional benefit is realized when the product is heated because the pH of the composition, and in turn the product, goes down giving the added potential of  
25 the composition of destroying any micro-organisms, their toxins or other harmful substances.

The composition of the present invention was found to be a "preservative." The composition is not corrosive; however, it can create an environment where destructive  
30 micro-organisms cannot live and propagate. The utility of this method of preservation is that additional chemicals do not have to be added to the food or other substance to be

preserved because the inherent low pH of the mixture is preservative. Since preservative chemicals do not have to be added to the food substance, taste is improved and residues are avoided. Organoleptic testing of a number of  
5 freshly preserved and previously preserved food stuffs have revealed the addition of composition improves taste and eliminates preservative flavors. The term "organoleptic" means making an impression based upon senses of an organ or the whole organism. In another use, the composition was  
10 added to various food dressing, fresh juices and fermented beverages (wine). The resulting taste was unanimously judged better than the starting or control beverage. Use of the composition both as a preservative and taste enhancer for food and beverages will produce a safer and  
15 more desirable product. Additionally the composition can be added to biologics, pharmaceuticals and other preservative sensitive products to enhance their safety and extend shelf life. It can also be used as an ingredient to adjust product pH.

20 Conventional cleaning of biopharmaceutical and vaccine equipment is always problematical. Bioreactor vessels, where genetically altered yeast and bacteria produce biopharmaceutical products, are very sensitive to residues left during the cleaning process. The adduct or composition  
25 of the present invention is extremely useful in the primary cleaning of these vessels following production termination and for final cleaning and rinsing just prior to reestablishing the culture in the reactor vessel. The composition's ability to completely remove residues will  
30 insure the success of the culture and eliminate the possibility of contamination in the biopharmaceutical or vaccine product.

Another field of manufacturing where cleaning is critical is in the precision injection molding of plastic and composite materials for critical use parts in medical devices and other industrial products. The composition of the present invention can clean the injection molds quickly and efficiently between runs without damaging the molds or leaving residues which can cause defects in the product. Additionally, the composition could be used to remove excess materials from the parts and acid etch or clean parts prior to assembly and welding. The composition of the present invention is useful to clean the surface of non metallic parts to be chemically, heat or ultrasonically welded. If the device is wet packaged, i.e. suture material, then the composition can be used as a packaging preservative.

Agricultural applications for the composition of the present invention are of special interest. The ability to manipulate the pH of hydroponic plant production water will influence fruit production and disease control. Synchronization of harvest and completeness of harvest can be aided by the composition. Olive, nut and some fruit trees are harvested by mechanical shaking. This shaking procedure must occur several times because the fruit and stem do not always ripen at the same time. Spraying the tree with the composition prior to harvest activities can cause the stems and produce to mature rapidly. Only one or two shaking procedures will be required to completely harvest the produce, thus reducing harvest cost and damage to the trees.

Bacteria, fungus, yeast and molds can reduce plant yields or effect the quality of crops near, at, or post harvest. The composition of the present invention can be

useful in preventing mold and mildew when crops in production are subjected to wet conditions. This is especially true in corn, maize and other grain sorgham production. Grapes destined for raisin production are  
5 harvested and left to dry in the field on paper or cloth tarps between the vines. If wet weather persists the raisins will mold during the drying process resulting in an unusable product. Spraying the composition on the grapes prior to harvest, dipping the clusters during harvest,  
10 treating the tarps, spraying the drying clusters, and washing the raisins prior to packing will result in raisins free of mold. The same methods can be used to assure uniformity of grapes during wine making. The composition of the present invention can be used to control pH and  
15 adjust taste of wine and other fermented beverages.

The same use of the composition of the present invention can be made when storing grains. Mold, mildew and other fungal infestations of stored grains produce mycotoxins. These mycotoxins are very harmful to animals  
20 that consume contaminated grains. Mycotoxin intoxication results in organ damage, decreased production, or death. Chemicals containing mercury and iodine are used to preserve planting seed, but there are no preservatives for grains destined for food or feed which do not leave harmful  
25 residues. Grains at harvest, during processing or in storage could be exposed to the composition, with or without additives, to create an environment where these organisms would not grow on the grain or in the storage container.

30 Specific field applications for military use are numerous. The primary application is in the decontamination of drinking water. Current methods for

individual drinking water decontamination consist of placing iodine tablets into a canteen of water and waiting a period of time. If a small amount of the composition of the present invention is added to the water, time for  
5 disinfection would be significantly reduced and there would be no need for iodine tablets. Additional applications for field living would include field waste decontamination, cooking liquid for food sources of questionable sanitary status, first aid irrigation solution for wounds and  
10 decontamination, dilution and clean up of toxic or dangerous substance spills, and equipment cleaning and decontamination. This is especially important when food service under field conditions does not always allow for hot water cleaning of equipment.

15 A biological contaminant is defined as a biological organism, or the product of biological organism, or both, each of which contaminates the environment and useful products. This biological contaminant results in making the environment or product hazardous.

20 Bacteria, fungi, mold, mildew, and viruses have potentially reactive substances in their cell wall/membranes; however, they hide in cells (viruses and some bacteria) and/or secrete biofilms (most bacteria, fungi, mold and mildew) to protect them from the  
25 environment.

The outer covering, i.e. epidermis, of animals and cuticle of plants resist the growth and/or entry of the above microorganisms into the interior of the complex organism. One of the microbial growth prevention methods  
30 used by plants and animals is the maintenance of a surface pH or secretion of a coating that is not conducive to the attachment and propagation of micro-organisms. After a

plant product is harvested or an animal product processed, these products lose the ability to resist the infestation of micro-organisms. By spraying the AGIIS plus defined additives on fruits, vegetables, and whole plants post  
5 harvest or washing or packing animal products in the composition, the growth and propagation of micro-organisms in these products can be reduced. If plant or animal products are packed in the composition an additional benefit is realized when the product is heated because the  
10 pH of the composition, and in turn the product, goes down giving the added potential of the composition of destroying any micro-organisms, their toxins or other harmful substances.

The composition of the present invention was found to  
15 be a "preservative." The composition is not corrosive; however, it can create an environment where destructive micro-organisms cannot live and propagate, thus prolong the shelf-life of the product. The utility of this method of preservation is that additional chemicals do not have to be  
20 added to the food or other substance to be preserved because the inherent low pH of the mixture is preservative. Since preservative chemicals do not have to be added to the food substance, taste is improved and residues are avoided. Organoleptic testing of a number of freshly preserved and  
25 previously preserved food stuffs have revealed the addition of composition improves taste and eliminates preservative flavors. Organoleptic means making an impression based upon senses of an organ or the whole organism. In another use the composition was added to various fresh juices and  
30 fermented beverages (wine). The resulting taste was unanimously judged better than the starting or control beverage. Use of the composition both as a preservative

and taste enhancer for food and beverages will produce a safer and more desirable product. Additionally the composition can be added to biologics, pharmaceuticals and other preservative sensitive products to enhance their safety and extend shelf life. It can also be used as an ingredient to adjust product pH.

Some biofilms are resistant to the AGIIS; however, this resistance can be overcome by adding small amounts of polar organic molecules namely, the additive to the AGIIS. Molecules such as ethyl alcohol, lactic acid and surfactants (such as polysorbate 80, (Tween 80) can enhance the activity of the composition as well as increase the speed and efficacy of the antimicrobial properties of the composition.

In the composition of the present invention, the addition of one, two or all of the above mentioned classes of additives, to the aqueous solution of AGIIS appear to enhance the penetration or the contact of reactive surface between the composition and the cleaning target. Broadly, one embodiment of the present invention is a combination of the aqueous solution of AGIIS with ethyl alcohol, lactic acid, and polysorbate 80, also known as Tween 80. This combination not only cleans a surface of organic or inorganic residues, but kills up to  $10^8$  organisms. Another combination, when applied with heat, will remove bacterial endotoxins from glass, metal and plastic surfaces without damaging these surfaces. This capability lends the combinations to be useful in the cleaning of pharmaceutical or biological processing and manufacturing equipment "in place." This capability does not require the dismantling of this expensive equipment for cleaning. Reduction of endo-toxins on processing equipment and primary packaging



of vaccines and pharmaceutical products is a requirement for safe use of drugs and vaccines.

Conventional cleaning of biopharmaceutical and vaccine equipment is always problematical. Bioreactor vessels, where genetically altered yeast and bacteria produce biopharmaceutical products, are very sensitive to residues left during the cleaning process. The adduct or composition of the present invention is extremely useful in the primary cleaning of these vessels following production termination and for final cleaning and rinsing just prior to reestablishing the culture in the reactor vessel. The composition's ability to completely remove residues will insure the success of the culture and eliminate the possibility of contamination in the biopharmaceutical or vaccine product.

Another field of manufacturing where cleaning is critical is in the precision injection molding of plastic and composite materials for critical use parts in medical devices and other industrial products. The composition of the present invention can clean the injection molds quickly and efficiently between runs without damaging the molds or leaving residues which can cause defects in the product. Additionally, the composition could be used to remove excess materials from the parts and acid etch or clean parts prior to assembly and welding. The composition of the present invention is useful to clean the surface of non metallic parts to be chemically, heat or ultrasonically welded. Also, the adduct of the present invention is useful to remove excess adhesive materials and generally decontaminate the medical device prior to packaging. If the device is wet packaged, i.e. suture material, then the composition can be used as a packaging preservative.

Agricultural applications for the adduct or composition of the present invention are of special interest. The ability to manipulate the pH of hydroponic plant production water will influence fruit production and disease control. Synchronization of harvest and completeness of harvest can be aided by the composition. Olive, nut and some fruit trees are harvested by mechanical shaking. This shaking procedure must occur several times because the fruit and stem do not always ripen at the same time. Spraying the tree with the composition prior to harvest activities can cause the stems and produce to mature rapidly. Only one or two shaking procedures will be required to completely harvest the produce, thus reducing harvest cost and damage to the trees.

Bacteria, fungus, yeast and molds can reduce plant yields or effect the quality of crops near, at, or post harvest. The adduct or the composition of the present invention can be useful in preventing mold and mildew when crops in production are subjected to wet conditions. This is especially true in corn, maize and other grain sorgham production. Grapes destined for raisin production are harvested and left to dry in the field on paper or cloth tarps between the vines. If wet weather persists the raisins will mold during the drying process resulting in an unusable product. Spraying the composition on the grapes prior to harvest, dipping the clusters during harvest, treating the tarps, spraying the drying clusters, and washing the raisins prior to packing will result in raisins free of mold. The same methods can be used to assure uniformity of grapes during wine making. The composition of the present invention can be used to control pH and adjust taste of wine and other fermented beverages.

The same use of the composition of the present invention can be made when storing grains. Mold, mildew and other fungal infestations of stored grains produce mycotoxins. These mycotoxins are very harmful to animals  
5 that consume contaminated grains. Mycotoxin intoxication results in organ damage, decreased production, or death. Chemicals containing mercury and iodine are used to preserve planting seed, but there are no preservatives for grains destined for food or feed which do not leave harmful  
10 residues. Grains at harvest, during processing or in storage could be exposed to the composition, with or without additives, to create an environment where these organisms would not grow on the grain or in the storage container.

15 Specific field applications for military use are numerous. The primary application is in the decontamination of drinking water. Current methods for individual drinking water decontamination consist of placing iodine tablets into a canteen of water and waiting  
20 a period of time. If a small amount of the adduct or the composition of the present invention is added to the water, time for disinfection would be significantly reduced and there would be no need for iodine tablets. Additional applications for field living would include field waste  
25 decontamination, cooking liquid for food sources of questionable sanitary status, first aid irrigation solution for wounds and decontamination, dilution and clean up of toxic or dangerous substance spills, and equipment cleaning and decontamination. This is especially important when  
30 food service under field conditions does not always allow for hot water cleaning of equipment.

The following examples are provided to further illustrate this invention and the manner in which it may be carried out. It will be understood, however, that the specific details given in the examples have been chosen for purposes of illustration only and not be construed as limiting the invention. Unless otherwise defined, the amount of each ingredient or component of the present invention is based on the weight percent of the final composition.

10

#### Example 1

AGIIS having an acid normality of 1.2 to 1.5 was prepared by the method of  $\text{H}_2\text{SO}_4/\text{Ca}(\text{OH})_2$ .

An amount of 1055 ml (19.2 moles, after purity adjustment and taking into account the amount of acid neutralized by base) of concentrated sulfuric acid (FCC Grade, 95-98% purity) was slowly added with stirring, to 16.868 L of RO/DI water in each of reaction flasks a, b, c, e, and f. The amount of water had been adjusted to allow for the volume of acid and the calcium hydroxide slurry. The mixture in each flask was mixed thoroughly. Each of the reaction flasks was chilled in an ice bath and the temperature of the mixture in the reaction flask was about 8-12°C. The mixture was continuously stirred at a rate of about 700 rpm.

Separately, a slurry was made by adding RO/DI water to 4 kg of calcium hydroxide (FCC Grade, 98% purity) making a final volume of 8 L. The mole ratio of calcium hydroxide to concentrated sulfuric acid was determined to be 0.45 to 1 from Figure 1. The slurry was a 50% (W/V) mixture of calcium hydroxide in water. The slurry was mixed well with a high-shear-force mixer until the slurry appeared uniform.

The slurry was then chilled to about 8-12°C in an ice bath and continuously stirred at about 700 rpm.

To each of the reaction flasks was added 150 ml of the calcium hydroxide slurry every 20 minutes until 1.276 L  
5 (i.e. 638 g dry weight, 8.61 moles, of calcium hydroxide) of the slurry had been added to each reaction vessel. The addition was again accompanied by well mixing at about 700 rpm.

After the completion of the addition of the calcium  
10 hydroxide to the reaction mixture in each reaction vessel, the mixture was filtered through a 5-micron filter.

The filtrate was allowed to sit for 12 hours, the clear solution was decanted to discard any precipitate formed. The resulting product was AGIIS having an acid  
15 normality of 1.2-1.5.

#### Example 2

AGIIS having an acid normality of 2 was prepared by  
20 the method of  $\text{H}_2\text{SO}_4/\text{Ca}(\text{OH})_2/\text{CaSO}_4$ .

For the preparation of 1 L of 2 N AGIIS, an amount of 79.54 ml (1.44 moles, after purity adjustment and taking into account the amount of acid to be neutralized by base) of concentrated sulfuric acid (FCC Grade, 95-98% purity)  
25 was slowly added, with stirring, to 853.93 ml of RO/DI water in a 2 L reaction flask. Five gram of calcium sulfate (FCC Grade, 95% purity) was then added slowly and with stirring to the reaction flask. The mixture was mixed thoroughly. At the point, the mixture would usually  
30 indicated an acid normality of 2.88. The reaction flask was chilled in an ice bath and the temperature of the mixture in the reaction flask was about 8-12°C. The

mixture was continuously stirred at a rate of about 700 rpm.

Separately, a slurry was made by adding 49.89 ml of RO/DI water to 33.26 g (0.44 mole, after purity adjustment) of calcium hydroxide (FCC Grace, 98% purity) making a final volume of 66.53 ml. The mole ratio of calcium hydroxide to concentrated sulfuric acid was determined to be 0.44 to 1 from Figure 1. The slurry was mixed well with a high-shear-force mixer until the slurry appeared uniform. The slurry was then chilled to about 8-12°C in an ice bath and continuous stirred at about 700 rpm.

The slurry was then slowly added over a period of 2-3 hours to the mixture, still chilled in an ice bath and being stirred at about 700 rpm.

After the completion of the addition of slurry to the mixture, the product was filtered through a 5-micron filter. It was normal to observe a 20% loss in volume of the mixture due to the retention of the solution by the salt and removal of the salt.

The filtrate was allow to sit for 12 hours, the clear solution was decanted to discard any precipitate formed. The resulting product was AGIIS having an acid normality of 2.

### Example 3

AGIIS having an acid normality of 12 was prepared by the method of  $\text{H}_2\text{SO}_4/\text{Ca}(\text{OH})_2/\text{CaSO}_4$ .

For the preparation of 1 L of 12 N AGIIS, an amount of 434.17 ml (7.86 moles, after purity adjustment and taking into account amount of acid neutralized by base) of concentrated sulfuric acid (FCC Grade, 95-98% purity) was slowly added, with stirring, to 284.60 ml of RO/DI water in

a 2 L reaction flask. Three gram of calcium sulfate (FCC Grade, 95% purity) was then added slowly and with stirring to the reaction flask. The mixture was mixed thoroughly. The reaction flask was chilled in an ice bath and the  
5 temperature of the mixture in the reaction flask was about 8-12°C. The mixture was continuously stirred at a rate of about 700 rpm.

Separately, a slurry was made by adding 210.92 ml of RO/DI water to 140.61 g (1.86 moles, after purity  
10 adjustment) of calcium hydroxide (FCC Grace, 98% purity) making a final volume of 281.23 ml. The mole ratio of calcium hydroxide to concentrated sulfuric acid was determined to be 0.31 from Figure 1. The slurry was mixed well with a high-shear-force mixer until the slurry  
15 appeared uniform. The slurry was then chilled to about 8-12°C in an ice bath and continuous stirred at about 700 rpm.

The slurry was then slowly added over a period of 2-3 hours to the mixture, still chilled in an ice bath and  
20 being stirred at about 700 rpm.

After the completion of the addition of slurry to the mixture, the product was filtered through a 5-micron filter. It was normal to observe a 20% loss in volume of the mixture due to the retention of the solution by the  
25 salt and removal of the salt.

The filtrate was allow to sit for 12 hours, the clear solution was decanted to discard any precipitate formed. The resulting product was AGIIS having an acid normality of  
2.

30

## Example 4

Test to determine if a pH 1.8 solution comprised of AGIIS and two additives (lactic acid and ethanol) could reduce contamination of alfalfa sprout seeds inoculated  
5 with E. coli 0157:H7.

A pH 1.8 solution comprised of AGIIS mixed with 0.05% lactic acid and 0.5% ethanol, was used to decontaminate alfalfa sprout seeds inoculated with  $1.1 \times 10^{13}$  colony forming unit ("CFU") of E. coli 0157:H7 at 35°C, 40°C and  
10 45°C, respectively.

Treatment with the adduct comprising AGIIS and the additives combination defined above at 35° C reduced the level of E. coli 0157:H7 contamination by >6 logs. Treatment at 40°C did not significantly increase  
15 decontamination relative to that at 35°C. However, treatment at 45°C decreased contamination by >8 logs, a significant difference.

## Example 5

20 Experiment to determine if a pH 1.8 solution comprised of AGIIS and three additives (lactic acid, ethanol and Tween 80) could decontaminate alfalfa sprout seeds inoculated with E. coli 0157:H7.

A pH 1.8 solution comprised of AGIIS, 0.05% lactic  
25 acid, 0.5% ethanol and 0.2% Tween 80, was used to decontaminate alfalfa sprout seeds inoculated with  $1.1 \times 10^{13}$  CFU of E. coli 0157:H7 at 35°C, 40°C and 45°C, respectively.

Treatment with AGIIS and the additives combination  
30 defined above at 35°C and 40°C reduced the level of E. coli 0157:H7 contamination by >6 logs. However, treatment at



45°C decreased contamination by >8 logs, a significant difference.

#### Example 6

5        Experiment to determine if a pH 1.8 solution comprised of AGIIS and three additives (lactic acid, ethanol and Tween 80) could decontaminate alfalfa sprout seeds inoculated with Salmonella spp.

10        A pH 1.8 solution comprised of AGIIS, 0.05% lactic acid, 0.5% ethanol and 0.2% Tween 80, was used to decontaminate alfalfa sprout seeds inoculated with  $1.1 \times 10^{13}$  CFU of Salmonella spp, at 35°C, 40°C and 45°C, respectively.

15        Treatment with AGIIS and the additives combination defined above at 35°C and 40°C reduced the level of Salmonella spp, contamination by >3 logs. However, treatment at 45°C decreased contamination by > 4 logs, a significant difference.

20

#### Example 7

Study to assess the effect of treatment at different temperatures and with a pH 1.8 solution comprised of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 on the level of aerobic microbes associated with alfalfa sprouts (produce) prior to final packaging.

25        It was demonstrated that the number of CFU per gram of sprouts was reduced by 99.99% when they were treated with a pH 1.8 solution comprised of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 at ~25°C for 15 min. From an organoleptic perspective there was no apparent effect on the sprouts. However, sprouts treated at 40°C appeared

limp, and were not as visually appealing as sprouts treated at room temperature.

#### Example 8

5 Study to assess the effect of treatment at different temperatures and with a pH 1.8 solution comprised of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 on the shelf life of alfalfa sprouts held at 25°C.

Treatment at 40°C for 15 min. made the sprouts whiter  
10 and cleaner looking, but the sprouts could not withstand the elevated temperature and degraded very fast. All samples within 36 hours were wilted and it was subsequently demonstrated that the Tween 80 additive adversely effected the organoleptic properties of alfalfa sprouts.

15

#### Example 9

Effect of the addition of a pH 1.8 solution comprised of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 on the microflora of ground beef.

20 The object of the study was to determine if a pH 1.8 solution comprised of AGIIS, 0.05% lactic acid, 0.5% ethanol and 0.2% Tween 80 mixed with ground beef (hamburger) could reduce and/or control the level of meat associated microflora.

25 No difference in the organoleptic properties of the treated and untreated hamburger could be discerned. However, when meat specimens were stomached and the microbe level of each specimen was determined, it was shown that meat treated with the AGIIS/additive solution had an  
30 associated microbe population 50% lower than that of saline treated meat. Thus, it was concluded that the

AGIIS/additive solution could reduce the level of microbe contamination and extend the shelf life of treated meat.

#### Example 10

5 Study to assess the ability of treatment at 45°C with a pH 1.4 solution comprised of AGIIS, 0.05% lactic acid, 0.5% ethanol, and 0.2% Tween 80 to reduce mold/fungal contamination and growth on raisins.

It was demonstrated that treatment reduced the amount  
10 of mold/fungal contamination associated with the raisins and appeared to suppress further growth. From an organoleptic perspective treatment removed significantly more dirt and debris than washing with water and the treated raisins were more appealing in that they appeared  
15 darker and fresher.

#### Example 11

Effect of treatment 45°C with a pH 1.4 solution comprised of AGIIS, 0.05% lactic acid, 0.5% ethanol, and  
20 0.2% Tween 80 on the viability of E. coli 0157:H7 organisms.

E coli 0157:H7 organisms  $1 \times 10^7$  were treated with the pH 1.4 solution comprised of AGIIS, 0.05% lactic acid, 0.5% ethanol, and 0.2% Tween 80 at 45°C. Serial dilutions of the  
25 treated material were plated to determine the effect of the treatment on the viability of the E. coli 0157:H7 organisms. The treatment killed 100% of the organisms, i.e., the CFU/ml was reduced from  $1 \times 10^7$  to zero.

30

#### Example 12

Study to assess the effect of treatment with solutions comprised of AGIIS, 0.5% ethanol and 0.05% lactic acid

having a pH ranging from 1.4-1.8 on the shelf life of alfalfa sprouts held at 25°C.

Sprouts were treated for 10 min. in the containers in which the sprouts were purchased. With respect to organoleptic properties after 12-16 hours the deionized water and non-treated sprouts had a distinct sour smell to them. It was concluded that adequate amounts of water were not removed from the sprouts, therefore, they degraded quickly. It was further noted that Tween 80 was a negative factor in the extension of sprout shelf life. It was also observed that sprouts treated with pH 1.8 or lower were bleached. Despite the rotten and/or degraded appearance, sprouts treated with the prononium solutions did not have a rotten sour smell.

15

#### Example 13

Study to assess the effect of treatment with solutions comprised of AGIIS, 0.5% ethanol, 0.05% lactic acid and/or Tween 80 having a pH ranging from 1.4-1.8 on the shelf life of alfalfa sprouts held at 25°C.

Control sprouts and sprouts treated with deionized water did not appear as fresh as sprouts treated with the AGIIS. Control sprouts and sprouts treated with deionized water were browned and somewhat discolored, whereas AGIIS treated (30 seconds) sprouts looked fresher and whiter. After 24 hours sprouts treated with the lower pH AGIIS solutions (<1.8) exhibited excessive bleaching. At 48 hours post-treatment the control and deionized water treated sprouts were rotten and smelled badly (a lot of gas was produced by the samples). It was concluded that a pH 1.8 AGIIS containing solutions along with the additives ethanol (0.5%) and lactic acid (0.05%) were the most

effective in extending shelf life. The sprouts looked as fresh as the day they were purchased 72 hr post-treatment at room temperature. Thus, pH 1.8 solutions containing AGIIS, and 0.5% ethanol and 0.05% lactic acid can extend  
5 the shelf life of the alfalfa sprouts.

#### Example 14

Study to show that a pH 1.8 solution of AGIIS and 0.5% ethanol and 0.05% lactic acid kills E. coli used to  
10 contaminate sprout seed as opposed to physically removing the contaminants.

Seed samples (25 g each) were contaminated with either  $1 \times 10^6$  or  $1 \times 10^{13}$  E. coli 0157:H7 CFU. Seed samples were treated for 10 min with a pH 1.8 solution containing AGIIS  
15 and 0.5% ethanol and 0.05% lactic acid. A 1 mL aliquot of each seed suspension was removed and serially diluted following treatment. After plating each serial dilution the number of CFU associated with the seed were enumerated. Treatment reduced the number of organisms associated with  
20 the seeds by 4-5 logs. The bacteria are killed not just washed off.

#### Example 15

Test to determine if treatment with a pH 1.8 solution  
25 of AGIIS, 0.5% ethanol, 0.05% lactic acid, and 0.2% Tween 80 could reduce and/or kill microbial organisms associated with the surface of tomatoes. The normal level of microbial contaminants associated with the surface of tomatoes was shown to be  $1 \times 10^6$ . After treatment, a 3-4 log reduction  
30 in surface associated organisms was demonstrated. A 3-4 log reduction is sufficient to provide for an extended

shelf life and reduce the probability of contamination of products made from tomatoes such as salsa.

#### Example 16

5       Studies pertaining to the effect of treatment with a pH 1.8 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 on microbial contamination and organoleptic properties.

10       One dozen chicken legs purchased from a local grocery were individually packaged and immersed for 10 min either in a pH 1.8 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80, or sterile saline (control). All chicken legs were then incubated at ambient room temperature for 24 hr.

15       The number of bacteria associated with drumsticks immersed in sterile saline after the 24 hr incubation period was  $\sim 7 \times 10^{10}$  as compared to  $\sim 6 \times 10^7$  for drumsticks treated with a pH 1.8 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80. With respect to the  
20       organoleptic properties, the drumsticks immersed in saline had a strong rotten smell after the 24 hr incubation period whereas the drumsticks treated with a pH 1.8 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 exhibited a fresh smell not unlike that of the drumsticks  
25       when purchased. Moreover, treatment maintained the fresh appearance of the chicken.

30       It was therefore concluded that treatment with a pH 1.8 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 could extend the shelf life of fresh meats, and in particular chicken.

## Example 17

Studies pertaining to the effect of treatment with a pH 1.8 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 on microbial contamination and organoleptic properties of shrimp.

Large headless shrimp purchased from a local grocery were packaged and immersed for 10 min either in a pH 1.8 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 or sterile saline (control). All shrimp were then incubated at ambient room temperature for 24 hr. The number of bacteria associated with shrimp immersed in sterile saline after the 24 hr incubation period was  $\sim 3.8 \times 10^{10}$  as compared to  $\sim 1.4 \times 10^9$  for shrimp treated with a pH 1.8 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80. With respect to the organoleptic properties, the treated and untreated shrimp did not differ.

In another experiment, the shelf-life (freshness and physical appearance) of whole shrimp was found to have increased from about 0.75 day to 1.5 days at 25° C.

## Example 18

Investigation of the capacity of a pH 1.8 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 to decontaminate chicken halves inoculated with E. coli 0157:H7.

Chicken halves inoculated with E. coli 0157:H7 were immersed in a pH 1.8 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 or sterile saline for 10 min. After immersion the halves were placed in plastic bags with culture media and incubated at 37°C for 2 hr. After the two

hour incubation period the E. coli 0157:H7 titers were determined.

It was demonstrated that treatment with a pH 1.8 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 reduced E coli 0157:H7 growth by ~1 log, i.e., from  $1.5 \times 10^9$  to  $1.2 \times 10^8$ . No difference in organoleptic properties were noted.

#### Example 19

10 Study to assess the ability of a pH 1.8 solution of AGIIS, 0.5% ethanol and 0.05% lactic acid to extend the shelf life of fruits and vegetables.

##### Berries:

Blueberry, Raspberry and Grapes:

15 Treatment with a pH 1.8 solution of AGIIS, 0.5% ethanol and 0.05% lactic acid did not prevent multiplication of berry associated fungi but berries retained their shape and exhibited an overall better appearance.  
Treatment had no effect on strawberries.

##### 20 Whole Melons:

Immersion of whole cantaloupe for 30 min in a pH 1.8 solution of AGIIS, 0.5% ethanol and 0.05% lactic acid slowed decay (observation made 48 hr post-treatment).

##### Cut Fruit:

25 Immersion of quarter sections of cantaloupe and honeydew melons in a pH 1.4 solution of AGIIS, 0.5% ethanol and 0.05% lactic acid suppress decay. The fruit treated with the pH 1.4 solution of AGIIS, 0.5% ethanol and 0.05% lactic acid refrigerated or kept at ambient room temperature  
30 retained their color and smelled fresh 24 hr later, whereas the fruit treated with sterile water had a rotten smell.

Cut Fruit Salad (mixture of apple, pear, peach and melons):



Cut fruit salad treated with a pH 1.4 solution of AGIIS, 0.5% ethanol and 0.05% lactic acid had a fresh appearance 2 hours (room temperature) after treatment whereas fruit salad treated with water appeared brown and in the process of decay.

Cut Lettuce:

Cut lettuce treated with a pH 1.8 solution of AGIIS, 0.5% ethanol and 0.05% lactic acid and incubated either at room temperature or at 40°C had no effect with respect to decay at the edge of the leaves. However, the leaves of lettuce treated with the pH 1.8 solution of AGIIS, 0.5% ethanol and 0.05% lactic acid were intact after incubation for 6 days at room temperature, whereas the water treated leaves were slimy and had decayed to a rotten mass.

15 Tomatoes:

Cherry tomatoes treated with a pH 1.8 solution of AGIIS, 0.5% ethanol and 0.05% lactic acid or water exhibited no difference in appearance. However, as in a previous experiment tomatoes immersed in AGIIS exhibited a significantly extended shelf life.

Example 20

The Ability of Adduct to clean Blueberry and Raspberry.

25 A combination composition, pH 1.4 solution of AGIIS, 0.5% ethyl alcohol and 0.05% lactic acid was applied to treat the berries by 15 minutes of immersion. A control group of berries treated in autoclaved Saline were performed parallel. Treated and non-treated berries were stomached with 1ml/g saline. A comparison of plate-counts between treated and non-treated group were made. As both

groups had a bacterial count number lower than  $10^3$ , it was concluded that the experiment is of no significance.

#### Example 21

5        Study to determine if treatment with a pH 1.2 solution of AGIIS, 0.5% ethanol and 0.05% lactic acid can reduce microbial contamination of cut fruit.

      The fruit of a cantaloupe and honeydew melon was cut into 1-1.5 inch cubes and inoculated for 10 min with  $2.5 \times 10^9$   
10    E.coli O157:H7 organisms suspended in saline. After 10 min the melon cubes were treated with a pH 1.2 solution of AGIIS, 0.5% ethanol and 0.05% lactic acid or sterile saline by immersion for 10 min. After treatment melon cubes were stomached and the number of associated bacteria were  
15    determined. The number of E. coli O157:H7 CFU associated with cantaloupe cubes was  $7.2 \times 10^3$  as compared to  $3.4 \times 10^5$  associated with saline treated cubes, i.e., treatment reduced the contamination level by ~98%. The number of E. coli O157:H7 CFU associated with honeydew cubes was  $5.3 \times$   
20     $10^3$  as compared to  $1.4 \times 10^5$  associated with saline treated cubes, i.e., treatment reduced the contamination level by ~95%.

#### Example 22

25        Study to determine if treatment with a solution of AGIIS, ethanol and lactic acid can prevent oxidation of potatoes. Potatoes were peeled and immersed in a pH 1.4 solution of AGIIS, 0.5% ethanol, 0.05% lactic acid and 0.2% Tween 80 or in water for 15 min. After treatment potatoes  
30    were removed and incubated on an open shelf for 24 hrs at ambient room temperature. Water treated potatoes started turning black within 30 min. whereas potatoes treated with

the AGIIS solution stayed white for several hours and a noticeable difference was evident 24 hr post-treatment.

#### Example 23

5 A previously painted surface on a house built in 1895 was cleaned with a solution of AGIIS, pH 2, and 0.2% Tween-80 surfactant. All surface contamination was removed and was visually cleaner than a corresponding area cleaned with an ammonia cleaner. The cleaned surface was allowed to dry  
10 and a coat of acrylic paint was applied. One week later the paint appeared normal and had not bubbled or peeled.

#### Example 24

A used driveway with oil stains was cleaned using  
15 AGIIS pH 2 and a 0.2% Tween-80 surfactant. Two identical areas were cleaned. The area treated with the AGIIS solution was whiter than the detergent only area and was judged cleaner by the observer.

#### Example 25

20 A small area of a new concrete sidewalk was sprayed with pH 1.8 AGIIS and 0.05% lactic acid mixture. After the area was allowed to dry the sidewalk was sealed with a concrete sealant. The appearance of the area etched with  
25 the AGIIS was different from the surrounding concrete; however, the sealer was adhered to the concrete and was impervious to water.

#### Example 26

30 A green pepper plant had 3 peppers on the plant at various stages of development. A mixture solution of pH 1.8 AGIIS and 0.5% lactic acid was sprayed on the plant

every other day for 6 consecutive applications. The largest of the peppers began to immediately turn red. The smaller peppers increased in size rapidly, but began turning red after the third application. At the end of the 5 treatments, the peppers were of different sizes, but uniformly red in color.

#### Example 27

Potable water contained non-coliform organisms. A 10 mixture of pH 1.4 AGIIS and 0.05% lactic acid was added to this water to bring the pH to 2.0. There was no growth when the water was cultured for organisms and the water could be consumed without adverse effects.

15

#### Example 28

Test to determine if a pH 1.1 solution comprised of AGIIS, 0.5% ethanol and 0.05% lactic acid can destroy the viability of bacteria present in water collected from a street puddle. Water was collected from a puddle at the 20 corner in front of a laboratory building. It was determined that the pH of the water was 7.4. Puddle water was titrated with a pH 1.1 solution comprised of AGIIS, 0.5% ethanol and 0.05% lactic acid or sterile saline and treated at ambient room temperature. Following treatment, 25 an aliquot of the AGIIS and saline treated water was serially diluted and plated to determine the number of viable organisms. The pH 1.1 solution comprised of AGIIS, 0.5% ethanol and 0.05% lactic acid used to treat the water effectively decreased the number of viable organism 30 relative to the control of saline.

## Example 29

Experiment to determine if a pH 1.56 solution comprised of AGIIS and three additives (lactic acid, ethanol and Tween 80) could decontaminate alfalfa sprout seeds inoculated with *Listeria monocytogenes*. A pH 1.56 solution comprised of AGIIS, 0.05% lactic acid, 0.5% ethanol and 0.2% Tween 80, was used to decontaminate alfalfa sprout seeds inoculated with *Listeria monocytogenes* at 45°C. Treatment with the adduct combination defined above at 45° C reduced the level of *Listeria monocytogenes* contamination by > 6 logs compared to sprout seeds inoculated with *Listeria monocytogenes* and treated with sterile saline.

## Example 30

The following protocol was used in this and the next three experiments.

1. Make a solution by adding concentrated AGIIS ( $\text{Ca}(\text{OH})_2/\text{H}_2\text{SO}_4/\text{CaSO}_4$  method) solution to deionized  $\text{H}_2\text{O}$  to adjust the pH to desired level, and then per-acetic acid was added.
2. Culture bacteria until just before they reach stationary phase.
3. Set out and label numerically sterile 15ml Falcon tubes. Add 4 ml of solution prepared as described in step #1 to the first tube. Add 4 ml of sterile saline to all the rest of the tubes.
4. Set the timer to 60 seconds.
5. Add 1 ml of bacterial culture to first tube, start the timer.

6. After 60 sec has elapsed, immediately transfer 1 ml to the next tube. Vortex and transfer 1 ml to the next tube. Perform in succession to complete serial dilution.
- 5 7. Plate in triplicate by transferring 0.1 ml from each tube to three different culture plates. Incubate each plate overnight and count the number of colonies per plate 24 hr later to determine the number of survivors post-treatment.
- 10 8. To determine the number of bacteria subjected to treatment, make a serial dilution of original culture and plate in triplicate 0.1 ml from each dilution.
- 15 Tomatoes were contaminated with  $>1 \times 10^7$  CFU of salmonella by dipping the tomatoes in the bacteria and then letting them air dry. Treated tomatoes were immersed in a solution of an adduct containing AGIIS (pH 1.2, 0.063 N) mixed with 80 ppm of per-acetic acid for 1 min. After
- 20 treatment the tomatoes were stomached and an aliquot was plated to quantitate the number of associated organisms. In summary treatment with the adduct reduced the number of associated organisms by  $>4$  logs.

25 Example 31

Blueberries were contaminated with  $>1 \times 10^{10}$  CFU of salmonella by dipping the blueberries in the bacteria and then letting them air dry. Treated blueberries were immersed in a solution of an adduct containing AGIIS (pH 1.2, 0.063 N) mixed with 0.48ml/L, or 80 ppm, of per-acetic acid for 1 min. After treatment the blueberries were stomached and an aliquot was plated to quantitate the

number of associated organisms. In summary treatment with the adduct reduced the number of associated organisms by >5 logs.

5

## Example 32.

Apples were cut into quarters by cutting through the stem. The pieces were contaminated  $>1 \times 10^9$  CFU of salmonella by dipping the pieces in the bacteria and then letting them air dry. Treated apples were immersed in a solution of an adduct containing AGIIS (pH 1.2, 0.063 N) mixed with 0.48ml/L, or 80 ppm, of per-acetic acid for 1 min. After treatment the apples were rinsed vigorously with saline and an aliquot of the rinse was plated to quantitate the number of associated organisms. In summary treatment with the adduct reduced the number of associated organisms by >8 logs.

## Example 33

Broccoli were contaminated with  $>1 \times 10^7$  CFU of salmonella by dipping the broccoli in the bacteria and then letting them air dry. Treated broccoli was immersed in a solution of an adduct containing AGIIS (pH 1.2, 0.063 N) mixed with 0.48ml/L, or 80 ppm, of per-acetic acid for 1 min. After treatment the broccoli were stomachered and an aliquot was plated to quantitate the number of associated organisms. In summary treatment with the adduct reduced the number of associated organisms by >5 logs.

## WHAT IS CLAIMED IS:

1. A composition comprising:
  - 2 an acidic sparingly-soluble Group IIA complex
  - 3 ("AGIIS"); and
  - 4 an additive.
2. The composition of claim 1, wherein the AGIIS is
  - 2 isolated from a mixture comprising a mineral acid and a
  - 3 Group IIA hydroxide, or a Group IIA salt of a dibasic acid,
  - 4 or a mixture of the two.
3. The composition of claim 2, wherein the Group IIA
  - 2 hydroxide is calcium hydroxide, the mineral acid is
  - 3 sulfuric acid, and the Group IIA salt of a dibasic acid is
  - 4 calcium sulfate.
4. The composition of claim 3, wherein the AGIIS
  - 2 having a certain acid normality is less effective in
  - 3 charring sucrose and less corrosive to an animal skin than
  - 4 a saturated solution of calcium sulfate in sulfuric acid
  - 5 having the same acid normality, and wherein the AGIIS is
  - 6 non-volatile at room temperature and pressure.
5. The composition of claim 1, wherein the additive
  - 2 comprises an alcohol.
6. The composition of claim 1, wherein the additive
  - 2 comprises an organic acid.



7. The composition of claim 1, wherein the additive  
2 comprises a surfactant.

8. The composition of claim 1, wherein, based on the  
2 final weight of the composition, the amount of AGIIS ranges  
3 from about 0.01 % to about 99.99 % of a AGIIS, and the  
4 amount of the additive ranges from about 0.01 % to about  
5 99.99 % of an additive.

9. A composition comprising:  
2 AGIIS prepared by mixing calcium hydroxide and  
3 sulfuric acid with or without the addition of calcium  
4 sulfate; and  
5 an additive is an alcohol, an organic acid, a  
6 surface active agent, or a mixture thereof.

10. The composition of claim 9, wherein the sulfuric  
2 acid contains a predetermined amount of calcium sulfate.

11. The composition of claim 9, wherein the alcohol  
2 comprises a lower aliphatic alcohol having six or less  
3 carbon atoms.

12. The composition of claim 9, wherein the organic  
2 acid is lactic acid, acetic acid, propionic acid, oxalic  
3 acid, per-acetic acid, and periodic acid, periodic acid, or  
4 a mixture thereof.

13. The composition of claim 9, wherein the surface  
2 active agent is cationic, anionic, non-ionic or a mixture  
3 thereof.

14. The composition of claim 9, wherein the AGIIS  
2 having a certain acid normality is less effective in  
3 charring sucrose and less corrosive to an animal skin than  
4 a saturated solution of calcium sulfate in sulfuric acid  
5 having the same acid normality, and wherein the AGIIS is  
6 non-volatile at room temperature and pressure.

15. The composition of claim 9, wherein for every  
2 mole of sulfuric acid used, the amount of calcium hydroxide  
3 ranges from about 0.1 mole to about 0.5 mole.

16. The composition of claim 9, wherein, based on the  
2 final weight of the composition, the amount of AGIIS ranges  
3 from about 0.01 % to about 99.99 % of a AGIIS, and the  
4 amount of the additive ranges from about 0.01 % to about  
5 99.99 % of an additive.

17. A method for decreasing pH of a solution,  
2 comprising:  
3 adding to the solution an effective amount of a  
4 mixture comprising AGIIS and an additive.

18. A method for decreasing pH of a mixture  
2 comprising a AGIIS and an additive, the method comprising:  
3 heating the mixture.

19. A method for reducing biological contaminants in  
2 a plant product comprising:  
3 contacting the plant product with a mixture  
4 comprising AGIIS and an additive.

20. The method of claim 19, wherein the plant product  
2 is fresh fruit, a vegetable produce, a harvested plant, a  
3 seed, a preserved plant, or a packaged plant.

21. A method for reducing biological contaminants in  
2 an animal meat product comprising:  
3           contacting the animal meat product with a mixture  
4 comprising a AGIIS and an additive.

22. The method of claim 21, wherein the animal meat  
2 product is an animal prior to slaughter, an animal carcass  
3 prior to division, a divided and processed animal carcass,  
4 a dried animal product, a smoked animal product, a cured  
5 animal product, or an aged animal product.

23. A method for reducing biological contaminants in  
2 an equipment comprising:  
3           contacting the equipment with an aqueous mixture  
4 comprising AGIIS and an additive.

24. The method of claim 23, wherein the equipment is  
2 a plant harvesting equipment, a plant packaging equipment,  
3 an animal slaughter equipment, an animal carcass processing  
4 equipment, an equipment for dividing and processing an  
5 animal carcass, or an equipment for treating an animal  
6 product.

25. The method of claim 23, wherein the equipment is  
2 a food service equipment, an injection molding equipment,  
3 a welding equipment, a packaging equipment, or an equipment  
4 requiring acid cleaning.

26. The method of claim 23, wherein the equipment is  
2 a medical instrument equipment.

27. A method for reducing biological contaminants in  
2 a packaging material comprising:

3                   contacting the packaging material with an aqueous  
4 mixture comprising AGIIS and an additive.

28. The method of claim 27, wherein the packaging  
2 material is a plant product packaging material or an animal  
3 product packaging material.

4

29. A method for preserving a consumable product,  
comprising:

                  contacting the consumable product with a mixture  
comprising AGIIS and an additive.

30. The method of claim 29, wherein the consumable  
2 product is a nutriment, a plant product, an animal product,  
3 a human medical product, a veterinary medical product, a  
4 biological product, a pharmaceutical product, or a medical  
5 device product.

31. A method for reducing the quantity of a  
2 biological toxin in a medium, comprising

3                   contacting the medium with a mixture comprising  
4 AGIIS and an additive.

32. The method of claim 31, wherein the medium is a  
2 nutriment, an equipment, a packaging material, water, or  
3 soil.

33. The method of claim 32, wherein the nutriment is  
2 food, feed, drink, food supplement, feed supplement, drink  
3 supplement, food dressing, pharmaceutical, biological  
4 product, seasoning, spices, flavoring agent, or stuffing.

34. The method of claim 31, wherein the toxin is an  
2 animal toxin, a bacterial toxin, a botulinus toxin, a  
3 cholera toxin, shiga like toxin, a streptococcus  
4 erythrogenic toxin, a dinoflagellate toxin, a diphtheria  
5 toxin, an erythrogenic toxin, an extracellular toxin, a  
6 fatigue toxin, an intracellular toxin, a scarlet fever  
7 erythrogenic toxin, or a Tunnickliff toxin.

35. The method of claim 31, wherein the toxin  
2 comprises an endotoxin.

36. The method of claim 31, wherein the toxin  
2 comprises a mycotoxin.

37. A method for reducing the quantity of spores in  
2 a medium, comprising  
3 contacting the medium with a mixture comprising  
4 AGIIS and an additive.

38. The method of claim 36, wherein the medium is a  
2 nutriment, an equipment, a packaging material, water, or  
3 soil.

39. The prepared nutriment of claim 38, wherein the  
2 nutriment is food, feed, drink, food supplement, feed  
3 supplement, drink supplement, food dressing,

4 pharmaceutical, biological product, seasoning, spices,  
5 flavoring agent, or stuffing.

40. A method for enhancing the bioavailability of a  
2 nutrient in a nutriment, comprising:  
3 adding to the nutriment a mixture comprising  
4 AGIIS and an additive.

41. The method of claim 40, wherein the nutrient is  
2 a carbohydrate, a protein, an enzyme, or an acid-stable  
3 vitamin.

42. The method of claim 40, wherein the nutriment is  
2 food, feed, drink, food supplement, feed supplement, drink  
3 supplement, food dressing, pharmaceutical, biological  
4 product, seasoning, spices, flavoring agent, or stuffing.

43. A method for incorporating a mixture comprising  
2 AGIIS and an additive into a nutriment, comprising:  
3 adding the mixture comprising the AGIIS and the  
4 additive to a suitable carrier to give a premixed product,  
5 and  
6 blending the premixed product with the nutriment.

44. The method of claim 43, wherein the suitable  
2 carrier is a methylcellulose, a psyllium, bran, rice hull  
3 or corn gluten.

45. The method of claim 43, wherein the nutriment is  
2 food, feed, drink, food supplement, feed supplement, drink  
3 supplement, food dressing, pharmaceutical, biological  
4 product, seasoning, spices, flavoring agent, or stuffing.

46. A method for cleaning a product, comprising:  
2                   contacting the product with a mixture comprising  
3 AGIIS and an additive.

47. The method of claim 46, wherein the product is a  
2 tissue, a microelectronic product, or a construction  
3 product.

48. The method of claim 47, wherein the construction  
2 product is new or reworked.

49. The method for extending shelf-life of nutriment,  
2 comprising:  
3                   contacting the nutriment with a mixture  
4 comprising a AGIIS and an additive.

50. The method of claim 49, wherein the nutriment is  
2 food, feed, drink, food supplement, feed supplement, drink  
3 supplement, food dressing, pharmaceutical, biological  
4 product, seasoning, spices, flavoring agent, or stuffing.

51. A method for synchronizing a harvest of a desired  
2 part of a plant, comprising:  
3                   contacting the desired part of the plant with a  
4 mixture comprising AGIIS and an additive.

52. A method for preserving or improving organoleptic  
2 quality of a desired part of a plant, comprising:  
3                   contacting the desired part of the plant with a  
4 mixture comprising AGIIS and an additive.

53. The method of claim 52, wherein the contacting of  
2 the desired part of the plant occurs prior to harvest,  
3 during harvest, during handling, or post harvest.

54. A method for preserving or improving organoleptic  
2 quality of a nutriment, comprising:  
3 contacting the nutriment with a mixture  
4 comprising AGIIS and an additive.

55. The method of claim 54, wherein the nutriment  
2 material is food, feed, drink, food supplement, feed  
3 supplement, drink supplement, food dressing,  
4 pharmaceutical, biological product, seasoning, spices,  
5 flavoring agent, or stuffing.

56. A method for reducing biological contaminations  
2 in water, comprising:  
3 adding to the water a sufficient amount of a mixture  
4 comprising AGIIS and an additive to reduce the biological  
5 contamination.

57. The method of claim 56, wherein the water is  
2 portable water, storm sewer water, or sanitary sewer water.

58. A method for preparing an adduct having a desired  
2 final acid normality, comprising:

3 (a) determining the amount of a mineral acid  
4 needed by the following equation:

5 
$$E_1 = (N/2) + (N/2 + B)$$

6 wherein  $E_1$  is the amount of the mineral acid,  
7 in moles, required before making purity adjustment; N is  
8 the desired final acid normality; and B is the mole ratio



9 of a Group IIA hydroxide to the mineral acid needed to  
10 obtain the AGIIS having N, and B is derived from a pre-  
11 plotted curve depicting the relationship of the mineral  
12 acid and the Group IIA hydroxide for a desired N;

13 (b) making purity adjustment for the mineral  
14 acid used by the following equation:

$$15 \quad E_2 = E_1 / C$$

16 wherein  $E_2$  is the amount of the mineral acid,  
17 in moles, required after purity adjustment;  $E_1$  is as defined  
18 above; and C is the purity adjustment factor for the  
19 mineral acid;

20 (c) determining the amount of water, in ml,  
21 needed to be added to the mineral acid by the following  
22 equation:

$$23 \quad G = J - E_2 - I$$

24 wherein G is the amount of water, in ml,  
25 required to be added to the mineral acid; J is the final  
26 volume of aqueous mineral acid solution; I is the volume  
27 amount of Group IIA hydroxide needed, given below; and  $E_2$  is  
28 as defined above;

29 (d) adding G to  $E_2$  to give the final aqueous  
30 solution of the mineral acid, wherein both G and  $E_2$  are as  
31 defined above;

32 (e) determining the amount of Group IIA  
33 hydroxide, in moles, needed by the following equation:

$$34 \quad F_1 = N / 2 \times B$$

35 wherein  $F_1$  is the amount of Group IIA  
36 hydroxide, in moles, needed before making purity  
37 adjustment; and B and N are as defined above;

38 (f) making purity adjustment for the Group IIA  
39 hydroxide used by the following equation:

$$40 \quad F_2 = F_1 / D$$

41                    wherein  $F_2$  is the amount of the Group IIA  
42 hydroxide, in moles, required after purity adjustment;  $F_1$  is  
43 as defined above; and D is the purity adjustment factor for  
44 the Group IIA hydroxide;

45                    (g) determining the amount of water, in ml,  
46 needed to make the solution or slurry of Group IIA  
47 hydroxide by the following equation:

$$48 \qquad \qquad \qquad H = F_2 \times 1.5$$

49                    wherein H is the amount of water, in ml,  
50 needed to make the solution or slurry of Group IIA  
51 hydroxide; and  $F_2$  is as defined above;

52                    (h) determining the amount of the aqueous  
53 solution or slurry of Group IIA hydroxide, in ml, needed to  
54 be added to the aqueous solution of mineral acid to give  
55 the AGIIS with a desired final acid normality by the  
56 following equation:

$$57 \qquad \qquad \qquad I = F_2 \times 2$$

58                    wherein I is the amount of Group IIA  
59 hydroxide solution or slurry, in ml, needed; and  $F_2$  is as  
60 defined above;

61                    (i) adding H to  $F_2$  to give the final aqueous  
62 solution or slurry of Group IIA hydroxide, wherein both H  
63 and  $F_2$  are as defined above;

64                    (j) adding the final aqueous solution or slurry  
65 of Group IIA hydroxide of (i) to the final aqueous solution  
66 of mineral acid of (d);

67                    (k) allowing the final aqueous solution or  
68 slurry of Group IIA hydroxide and the final aqueous  
69 solution of mineral acid of (j) to react;

70                    (l) removing solid formed from (K) to give a  
71 final AGIIS solution; and

72 (m) adding a predetermined amount of an additive  
73 to the final AGIIS solution.

59. The method of claim 58 further comprising adding  
2 a Group IIA salt of a dibasic acid to the final aqueous  
3 mineral acid solution of (d).

60. The method of claim 58, wherein the mineral acid  
2 is sulfuric acid, the Group IIA hydroxide is calcium  
3 hydroxide, and the Group IIA salt of a dibasic acid is  
4 calcium sulfate.

61. The adduct having a desired final acid normality  
2 prepared by the method of claim 58.

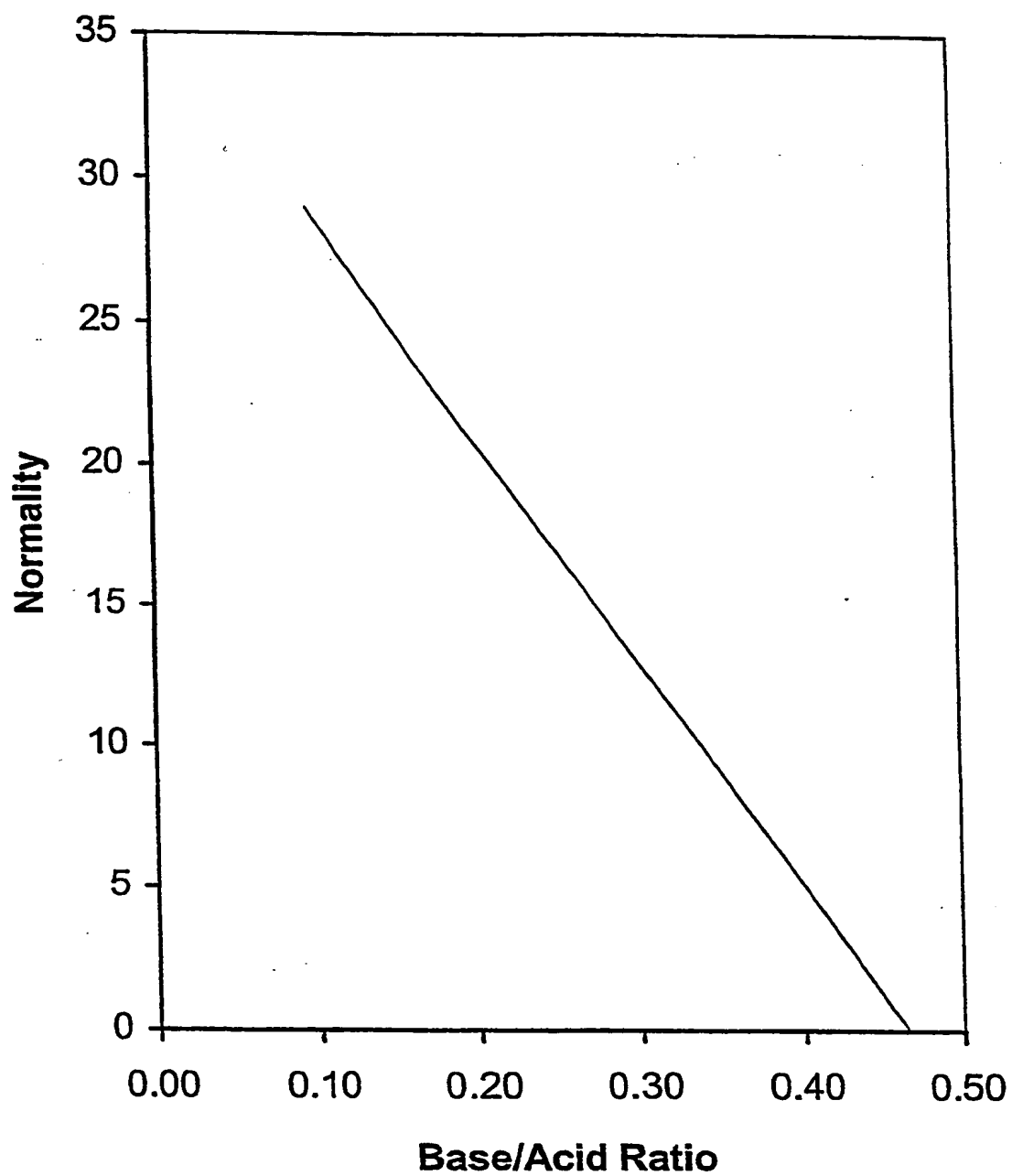


Fig. 1

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(US). YAO, Yu [US/US]; 401 Rocky Point Drive #5,  
Roseville, CA 95678 (US). LEWIS, David, E. [US/US];  
816 3rd Avenue, Eau Claire, WI 54703 (US).

(21) International Application Number: PCT/US00/03961

(74) Agent: CHWANG, T., Ling; Hitt Chwang & Gaines,  
P.C., Suite 225, 275 West Campbell Road, Richardson, TX  
75080 (US).

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(71) Applicant (*for all designated States except US*): MORN-  
INGSTAR DIAGNOSTICS [US/US]; Suite 130, 1376  
Lead Hill Boulevard, Roseville, CA 95661 (US).

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(72) Inventors; and

(75) Inventors/Applicants (*for US only*): KEMP, Maurice,  
Clarence [US/US]; 4401 Woodthrush Drive, El Dorado  
Hills, CA 95762 (US). LALUM, Robert, Blaine [US/US];  
6949 Greenback Lane #B, Citrus Heights, CA 95621 (US).  
XIE, Zhong, Wei [US/US]; 1141 Elderberry Circle,  
Folsom, CA 95630 (US). CUNHA, Michael, Anthony  
[US/US]; 355 Shadow Ridge Drive #3, Roseville, CA  
95678 (US). CARPENTER, Robert, H. [US/US]; 1303  
Pecan Street, Bastrop, TX 78602 (US). ZHANG, Shu  
[US/US]; 401 Rocky Point Drive #5, Roseville, CA 95678

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(54) Title: ADDUCT HAVING AN ACIDIC SOLUTION OF SPARINGLY-SOLUBLE GROUP IIA COMPLEXES

(57) Abstract: An adduct that has an acidic solution of sparingly-soluble Group IIA complexes ("AGIIS") and at least one additive. The AGIIS can be prepared by mixing a mineral acid (such as sulfuric acid), and a Group IIA hydroxide (such as calcium hydroxide) or a Group IIA salt of a dibasic acid (such as calcium sulfate), or a mixture of the two Group IIA compounds, followed by removing the solid formed. The additives can be an alcohol, an organic acid or a surface active agent. The composition has various uses, including cleaning, food production, decontamination, bioremediation, agricultural application, medical application, and detoxification of substances.

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# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 00/03961

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A23L3/358 A23L1/304 A61K33/08 A61K33/06 C01F11/46  
 //(A61K33/08,33:06,33:04)

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A23L A61K A23B C01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, PAJ, FSTA

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 264 017 A (VAN DE WALLE RICHARD H) 23 November 1993 (1993-11-23) column 1, line 11-19 column 2, line 60 -column 3, line 14 claims 1,12	1,2
P,X	WO 99 23896 A (NESTLE SA ;MALLANGI CHANDRASEKHARA REDDY (US); SHER ALEXANDER (US)) 20 May 1999 (1999-05-20) page 3, line 32 -page 4, line 6 page 4, line 29-34 page 5, line 19-33 page 6, line 19-25 claims 1,2,5,6,11-16	1,2,6,9, 12,17
	-/-	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

\* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

14 June 2000

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28. 08. 2000

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
 NL - 2280 HV Rijswijk  
 Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
 Fax: (+31-70) 340-3016

Authorized officer

Piret-Viprey, E

# INTERNATIONAL SEARCH REPORT

International Application No

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 90 05458 A (KINGSLEY I STEVEN) 31 May 1990 (1990-05-31)  page 6 page 7, paragraph 2 page 9, line 4	1,2,6, 17,19, 20, 29-33, 37-40, 42,49, 50,52-57
Y	examples I,II claims 1-5	58,61
Y	----- DATABASE WPI Derwent Publications Ltd., London, GB; AN 1989-003849 XP002140034 & JP 63 282117 A (SHIMIZU N.), 18 November 1988 (1988-11-18) abstract	58,61
X	----- WO 96 01566 A (DARATECH PTY LTD ;REYES VICENTE GERONIMO (AU)) 25 January 1996 (1996-01-25)  page 1, line 1-9 page 4, line 6 -page 5, line 21 claims 1,2,8,9,18,22,23,25,32,34 -----	1,2,19, 20, 29-33, 37-39, 49,50, 52-55

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US 00/03961

## Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☒ Claims Nos.:  
because they relate to subject matter not required to be searched by this Authority, namely:  
Although claim(s) are directed to a diagnostic method practised on the human/animal body, the search has been carried out and based on the alleged effects of the compound/composition.
2. ☒ Claims Nos.:  
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:  
see FURTHER INFORMATION sheet PCT/ISA/210
3. ☐ Claims Nos.:  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

## Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

R mark on Pr test

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.



## FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

## Continuation of Box I.2

Present claims 1,2 relate to an extremely large number of possible compounds. Support within the meaning of Article 6 PCT and/or disclosure within the meaning of Article 5 PCT is to be found, however, for only a very small proportion of the compounds claimed. In the present case, the claims so lack support, and the application so lacks disclosure, that a meaningful search over the whole of the claimed scope is impossible. Consequently, the search has been carried out for those parts of the claims which appear to be supported and disclosed, namely those parts relating to the compounds:

## - claim 3:

mineral acid= sulfuric acid

Group IIA hydroxide= calcium hydroxide

Group IIA salt of a dibasic acid= calcium sulfate

## - description: page 10, line 24 - page 11, line 20

mineral acid= sulfuric acid

Group IIA hydroxide= calcium hydroxide

Group IIA salt of a dibasic acid= calcium sulfate, calcium oxide, calcium carbonate.

## - examples: 1-3

mineral acid= sulfuric acid

Group IIA hydroxide= calcium hydroxide

Group IIA salt of a dibasic acid= calcium sulfate.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 00/03961

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
US 5264017	A	23-11-1993	CA	1318147 A	25-05-1993
WO 9923896	A	20-05-1999	US	6024994 A	15-02-2000
			AU	1152899 A	31-05-1999
WO 9005458	A	31-05-1990	NONE		
JP 63282117	A	18-11-1988	NONE		
WO 9601566	A	25-01-1996	AU	2916895 A	09-02-1996

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(74) Agent: **CHWANG, T., Ling**; Hitt Chwang & Gaines,  
P.C., Suite 225, 275 West Campbell Road, Richardson, TX  
75080 (US).

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(71) Applicant (*for all designated States except US*): **MORN-  
INGSTAR DIAGNOSTICS [US/US]**; Suite 130, 1376  
Lead Hill Boulevard, Roseville, CA 95661 (US).

(81) Designated States (*national*): AE, AL, AM, AT, AU, AZ,  
BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK,  
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IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU,  
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(AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU,  
MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM,  
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(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **KEMP, Maurice**,  
Clarence [US/US]; 4401 Woodthrush Drive, El Dorado  
Hills, CA 95762 (US). **LALUM, Robert, Blaine** [US/US];  
6949 Greenback Lane #B, Citrus Heights, CA 95621 (US).  
**XIE, Zhong, Wei** [US/US]; 1141 Elderberry Circle,  
Folsom, CA 95630 (US). **CUNHA, Michael, Anthony**  
[US/US]; 355 Shadow Ridge Drive #3, Roseville, CA  
95678 (US). **CARPENTER, Robert, H.** [US/US]; 1303  
Pecan Street, Bastrop, TX 78602 (US). **ZHANG, Shu**  
[US/US]; 401 Rocky Point Drive #5, Roseville, CA 95678  
(US). **YAO, Yu** [US/US]; 401 Rocky Point Drive #5,  
Roseville, CA 95678 (US). **LEWIS, David, E.** [US/US];  
816 3rd Avenue, Eau Claire, WI 54703 (US).

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- *With amended claims and statement.*

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22 February 2001

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(54) Title: **ADDUCT HAVING AN ACIDIC SOLUTION OF SPARINGLY-SOLUBLE GROUP IIA COMPLEXES**

(57) Abstract: An adduct that has an acidic solution of sparingly-soluble Group IIA complexes ("AGIIS") and at least one additive. The AGIIS can be prepared by mixing a mineral acid (such as sulfuric acid), and a Group IIA hydroxide (such as calcium hydroxide) or a Group IIA salt of a dibasic acid (such as calcium sulfate), or a mixture of the two Group IIA compounds, followed by removing the solid formed. The additives can be an alcohol, an organic acid or a surface active agent. The composition has various uses, including cleaning, food production, decontamination, bioremediation, agricultural application, medical application, and detoxification of substances.

WO 00/48469 A3

## AMENDED CLAIMS

[received by the International Bureau on 27 October 2000 (27.10.00);  
original claims 1, 2, 4, 8, 9, 12, 14, 17, 18, 19, 21, 23, 27, 29, 31, 37, 40, 43,  
46, 49, 51, 52, 54, 56 and 58 amended; remaining claims unchanged (12 pages)]

1. A composition comprising:
  - 2 a solution or suspension of an acidic sparingly-
  - 3 soluble Group IIA complex ("AGIIS"); and
  - 4 an additive.
2. The composition of claim 1, wherein the solution  
2 or suspension of the AGIIS is isolated from a mixture  
3 comprising a mineral acid and a Group IIA hydroxide, or a  
4 Group IIA salt of a dibasic acid, or a mixture of the two.
3. The composition of claim 2, wherein the Group IIA  
2 hydroxide is calcium hydroxide, the mineral acid is  
3 sulfuric acid, and the Group IIA salt of a dibasic acid is  
4 calcium sulfate.
4. The composition of claim 3, wherein the AGIIS  
2 having a certain acid normality is less effective in  
3 charring sucrose and less corrosive to an animal skin than  
4 a saturated solution of calcium sulfate in sulfuric acid  
5 having the same acid normality, and wherein the solution or  
6 suspension of an AGIIS is non-volatile at room temperature  
7 and pressure.
5. The composition of claim 1, wherein the additive  
2 comprises an alcohol.
6. The composition of claim 1, wherein the additive  
comprises an organic acid.

7. The composition of claim 1, wherein the additive  
2 comprises a surfactant.

8. The composition of claim 1, wherein, based on the  
2 final weight of the composition, the amount of the solution  
3 or suspension of the AGIIS ranges from about 0.01 % to  
4 about 99.99 % of the solution or suspension of the AGIIS,  
5 and the amount of the additive ranges from about 0.01 % to  
6 about 99.99 % of the additive.

9. A composition comprising:  
2 a solution or suspension of an AGIIS prepared by  
3 mixing calcium hydroxide and sulfuric acid with or without  
4 the addition of calcium sulfate; and  
5 an additive is an alcohol, an organic acid, a  
6 periodic acid, a surface active agent, or a mixture  
7 thereof.

10. The composition of claim 9, wherein the sulfuric  
2 acid contains a predetermined amount of calcium sulfate.

11. The composition of claim 9, wherein the alcohol  
2 comprises a lower aliphatic alcohol having six or less  
3 carbon atoms.

12. The composition of claim 9, wherein the organic  
2 acid is lactic acid, acetic acid, propionic acid, oxalic  
3 acid, per-acetic acid, or a mixture thereof.

13. The composition of claim 9, wherein the surface  
2 active agent is cationic, anionic, non-ionic or a mixture  
thereof.

14. The composition of claim 9, wherein the solution  
2 or suspension of the AGIIS having a certain acid normality  
3 is less effective in charring sucrose and less corrosive to  
4 an animal skin than a saturated solution of calcium sulfate  
5 in sulfuric acid having the same acid normality, and  
6 wherein the solution or suspension of an AGIIS is non-  
7 volatile at room temperature and pressure.

15. The composition of claim 9, wherein for every  
2 mole of sulfuric acid used, the amount of calcium hydroxide  
3 ranges from about 0.1 mole to about 0.5 mole.

16. The composition of claim 9, wherein, based on the  
2 final weight of the composition, the amount of the solution  
3 or suspension of AGIIS ranges from about 0.01 % to about  
4 99.99 % of the solution or suspension of the AGIIS, and the  
5 amount of the additive ranges from about 0.01 % to about  
6 99.99 % of the additive.

17. A method for decreasing pH of a solution,  
2 comprising:  
3           adding to the solution an effective amount of a  
4 mixture comprising a solution or suspension of an AGIIS and  
5 an additive.

18. A method for decreasing pH of a mixture  
2 comprising a solution or suspension of an AGIIS and an  
3 additive, the method comprising:  
4           heating the mixture.

19. A method for reducing biological contaminants in  
2 a plant product comprising:  
3           contacting the plant product with a mixture  
4 comprising a solution or suspension of an AGIIS and an  
additive.

20. The method of claim 19, wherein the plant product  
2 is fresh fruit, a vegetable produce, a harvested plant, a  
3 seed, a preserved plant, or a packaged plant.

21. A method for reducing biological contaminants in  
2 an animal meat product comprising:  
3           contacting the animal meat product with a mixture  
4 comprising a solution or suspension of an AGIIS and an  
5 additive.

22. The method of claim 21, wherein the animal meat  
2 product is an animal prior to slaughter, an animal carcass  
3 prior to division, a divided and processed animal carcass,  
4 a dried animal product, a smoked animal product, a cured  
5 animal product, or an aged animal product.

23. A method for reducing biological contaminants in  
2 an equipment comprising:  
3           contacting the equipment with an aqueous mixture  
4 comprising a solution or suspension of an AGIIS and an  
5 additive.

24. The method of claim 23, wherein the equipment is  
2 a plant harvesting equipment, a plant packaging equipment,  
3 an animal slaughter equipment, an animal carcass processing  
4 equipment, an equipment for dividing and processing an  
5 animal carcass, or an equipment for treating an animal  
6 product.

25. The method of claim 23, wherein the equipment is  
2 a food service equipment, an injection molding equipment,  
3 a welding equipment, a packaging equipment, or an equipment  
requiring acid cleaning.

26. The method of claim 23, wherein the equipment is  
2 a medical instrument equipment.

27. A method for reducing biological contaminants in  
2 a packaging material comprising:  
3       contacting the packaging material with an aqueous  
4 mixture comprising a solution or suspension of an AGIIS and  
5 an additive.

28. The method of claim 27, wherein the packaging  
2 material is a plant product packaging material or an animal  
3 product packaging material.  
4

29. A method for preserving a consumable product,  
comprising:  
      contacting the consumable product with a mixture  
comprising a solution or suspension of AGIIS and an  
additive.

30. The method of claim 29, wherein the consumable  
2 product is a nutriment, a plant product, an animal product,  
3 a human medical product, a veterinary medical product, a  
4 biological product, a pharmaceutical product, or a medical  
5 device product.

31. A method for reducing the quantity of a  
2 biological toxin in a medium, comprising  
3       contacting the medium with a mixture comprising  
4 a solution or suspension of an AGIIS and an additive.

32. The method of claim 31, wherein the medium is a  
2 nutriment, an equipment, a packaging material, water, or  
soil.



33. The method of claim 32, wherein the nutriment is  
2 food, feed, drink, food supplement, feed supplement, drink  
3 supplement, food dressing, pharmaceutical, biological  
4 product, seasoning, spices, flavoring agent, or stuffing.

34. The method of claim 31, wherein the toxin is an  
2 animal toxin, a bacterial toxin, a botulinus toxin, a  
3 cholera toxin, shiga like toxin, a streptococcus  
4 erythrogenic toxin, a dinoflagellate toxin, a diphtheria  
5 toxin, an erythrogenic toxin, an extracellular toxin, a  
6 fatigue toxin, an intracellular toxin, a scarlet fever  
7 erythrogenic toxin, or a Tunnickliff toxin.

35. The method of claim 31, wherein the toxin  
2 comprises an endotoxin.

36. The method of claim 31, wherein the toxin  
2 comprises a mycotoxin.

37. A method for reducing the quantity of spores in  
2 a medium, comprising  
3 contacting the medium with a mixture comprising  
4 a solution or suspension of an AGIIS and an additive.

38. The method of claim 36, wherein the medium is a  
2 nutriment, an equipment, a packaging material, water, or  
3 soil.

39. The prepared nutriment of claim 38, wherein the  
2 nutriment is food, feed, drink, food supplement, feed  
3 supplement, drink supplement, food dressing,

4 pharmaceutical, biological product, seasoning, spices,  
5 flavoring agent, or stuffing.

40. A method for enhancing the bioavailability of a  
2 nutrient in a nutriment, comprising:  
3 adding to the nutriment a mixture comprising a  
4 solution or suspension of an AGIIS and an additive.

41. The method of claim 40, wherein the nutrient is  
2 a carbohydrate, a protein, an enzyme, or an acid-stable  
3 vitamin.

42. The method of claim 40, wherein the nutriment is  
2 food, feed, drink, food supplement, feed supplement, drink  
3 supplement, food dressing, pharmaceutical, biological  
4 product, seasoning, spices, flavoring agent, or stuffing.

43. A method for incorporating a mixture comprising  
2 a solution or suspension of an AGIIS and an additive into  
3 a nutriment, comprising:  
4 adding the mixture comprising the solution or  
5 suspensions of the AGIIS and the additive to a suitable  
6 carrier to give a premixed product, and  
7 blending the premixed product with the nutriment.

44. The method of claim 43, wherein the suitable  
2 carrier is a methylcellulose, a psyllium, bran, rice hull  
3 or corn gluten.

45. The method of claim 43, wherein the nutriment is  
2 food, feed, drink, food supplement, feed supplement, drink  
3 supplement, food dressing, pharmaceutical, biological  
product, seasoning, spices, flavoring agent, or stuffing.

46. A method for cleaning a product, comprising:  
2           contacting the product with a mixture comprising  
3 a solution or suspension of an AGIIS and an additive.

47. The method of claim 46, wherein the product is a  
2 tissue, a microelectronic product, or a construction  
3 product.

48. The method of claim 47, wherein the construction  
2 product is new or reworked.

49. The method for extending shelf-life of nutriment,  
2 comprising:  
3           contacting the nutriment with a mixture  
4 comprising a solution or suspension of an AGIIS and an  
5 additive.

50. The method of claim 49, wherein the nutriment is  
2 food, feed, drink, food supplement, feed supplement, drink  
3 supplement, food dressing, pharmaceutical, biological  
4 product, seasoning, spices, flavoring agent, or stuffing.

51. A method for synchronizing a harvest of a desired  
2 part of a plant, comprising:  
3           contacting the desired part of the plant with a  
4 mixture comprising a solution or suspension of an AGIIS and  
5 an additive.

52. A method for preserving or improving organoleptic  
2 quality of a desired part of a plant, comprising:  
3           contacting the desired part of the plant with a  
4 mixture comprising a solution or suspension of an AGIIS and  
an additive.

53. The method of claim 52, wherein the contacting of  
2 the desired part of the plant occurs prior to harvest,  
3 during harvest, during handling, or post harvest.

54. A method for preserving or improving organoleptic  
2 quality of a nutriment, comprising:  
3 contacting the nutriment with a mixture  
4 comprising a solution or suspension of an AGIIS and an  
5 additive.

55. The method of claim 54, wherein the nutriment  
2 material is food, feed, drink, food supplement, feed  
3 supplement, drink supplement, food dressing,  
4 pharmaceutical, biological product, seasoning, spices,  
5 flavoring agent, or stuffing.

56. A method for reducing biological contaminations  
2 in water, comprising:  
3 adding to the water a sufficient amount of a mixture  
4 comprising a solution or suspension of an AGIIS and an  
5 additive to reduce the biological contamination.

57. The method of claim 56, wherein the water is  
2 portable water, storm sewer water, or sanitary sewer water.

58. A method for preparing an adduct having a desired  
2 final acid normality, comprising:

(a) determining the amount of a mineral acid  
needed by the following equation:

$$E_1 = (N/2) + (N/2 + B)$$

wherein  $E_1$  is the amount of the mineral acid,  
7 in moles, required before making purity adjustment; N is  
the desired final acid normality; and B is the mole ratio

9 of a Group IIA hydroxide to the mineral acid needed to  
10 obtain a solution or suspension of an AGIIS having N, and  
11 B is derived from a pre-plotted curve depicting the  
12 relationship of the mineral acid and the Group IIA  
13 hydroxide for a desired N;

14 (b) making purity adjustment for the mineral  
15 acid used by the following equation:

16 
$$E_2 = E_1/C$$

17 wherein  $E_2$  is the amount of the mineral acid,  
18 in moles, required after purity adjustment;  $E_1$  is as defined  
19 above; and C is the purity adjustment factor for the  
20 mineral acid;

21 (c) determining the amount of water, in ml,  
22 needed to be added to the mineral acid by the following  
23 equation:

24 
$$G = J - E_2 - I$$

25 wherein G is the amount of water, in ml,  
26 required to be added to the mineral acid; J is the final  
27 volume of aqueous mineral acid solution; I is the volume  
28 amount of Group IIA hydroxide needed, given below; and  $E_2$  is  
29 as defined above;

30 (d) adding G to  $E_2$  to give the final aqueous  
31 solution of the mineral acid, wherein both G and  $E_2$  are as  
32 defined above;

33 (e) determining the amount of Group IIA  
34 hydroxide, in moles, needed by the following equation:

35 
$$F_1 = N/2 \times B$$

36 wherein  $F_1$  is the amount of Group IIA  
37 hydroxide, in moles, needed before making purity  
38 adjustment; and B and N are as defined above;

39 (f) making purity adjustment for the Group IIA  
40 hydroxide used by the following equation:

41 
$$F_2 = F_1/D$$

41                   wherein  $F_2$  is the amount of the Group IIA  
42 hydroxide, in moles, required after purity adjustment;  $F_1$  is  
43 as defined above; and D is the purity adjustment factor for  
44 the Group IIA hydroxide;

45                   (g) determining the amount of water, in ml,  
46 needed to make the solution or slurry of Group IIA  
47 hydroxide by the following equation:

$$48 \qquad \qquad \qquad H = F_2 \times 1.5$$

49                   wherein H is the amount of water, in ml,  
50 needed to make the solution or slurry of Group IIA  
51 hydroxide; and  $F_2$  is as defined above;

52                   (h) determining the amount of the aqueous  
53 solution or slurry of Group IIA hydroxide, in ml, needed to  
54 be added to the aqueous solution of mineral acid to give  
55 the solution or suspension of the AGIIS with a desired  
56 final acid normality by the following equation:

$$57 \qquad \qquad \qquad I = F_2 \times 2$$

58                   wherein I is the amount of Group IIA  
59 hydroxide solution or slurry, in ml, needed; and  $F_2$  is as  
60 defined above;

61                   (i) adding H to  $F_2$  to give the final aqueous  
62 solution or slurry of Group IIA hydroxide, wherein both H  
63 and  $F_2$  are as defined above;

64                   (j) adding the final aqueous solution or slurry  
65 of Group IIA hydroxide of (i) to the final aqueous solution  
66 of mineral acid of (d);

67                   (k) allowing the final aqueous solution or  
68 slurry of Group IIA hydroxide and the final aqueous  
69 solution of mineral acid of (j) to react;

70                   (l) removing solid formed from (K) to give a  
71 final AGIIS solution or suspension; and

72 (m) adding a predetermined amount of an additive  
73 to the final a solution or suspension of an AGIIS solution  
74 or suspension.

75

59. The method of claim 58 further comprising adding  
2 a Group IIA salt of a dibasic acid to the final aqueous  
3 mineral acid solution of (d).

60. The method of claim 58, wherein the mineral acid  
2 is sulfuric acid, the Group IIA hydroxide is calcium  
3 hydroxide, and the Group IIA salt of a dibasic acid is  
4 calcium sulfate.

61. The adduct having a desired final acid normality  
prepared by the method of claim 58.

STATEMENT UNDER ARTICLE 19(1)

As amended, one aspect of Applicant's invention pertains to a composition that must be: (1) a solution or suspension; (2) acidic; (3) sparingly soluble Group IIA complex ("AGHS"); and (4) supplemented with an additive. The acidity comes from a mineral acid. To the extent removable, the  $\text{CaSO}_4$  formed is removed.

It is respectfully submitted that Applicant's claims are different and distinct from the teaching of the cited references discussed below:

A. U.S. Patent 5,264,017 teaches that the metal oxide or hydroxide used for the combination is in *excess* of the stoichiometric neutralization value. See, Col 2, line 60 to Col. 3, line 3. Thus, the combination is *basic, not acidic*. Further, the invention relates to agglomeration, pelletizing, or granulation. See, Col. 1, lines 11-18. It is *not a solution or suspension*.

B. WO 99 23896 teaches *neutralization the pH* of the complex. See, page 3, line 31, to page 4, line 6, and Claim 10. Thus the complex is *not acidic*. In fact, if the complex were acidic, it *cannot* be used to fortify milk, and would defeat the purpose of the invention. See, the Abstract.

C. WO 90 05458 teaches pickled foodstuffs and a method to pickle foodstuffs. The reference teaches using a *Group IA*, such as K and Na, (*not Group IIA*) salt. See, page 10, lines 16-18, and Example I on page 13. The acidulants used here are *organic acids*, not mineral acids. See, page 9, paragraph 6, and Claim 5.

D. XP002140034 teaches manufacturing *pure calcium sulfate crystals*, not a solution or suspension of an acidic sparingly-soluble Group IIA complex and an additive.

E. WO 96 01566 teaches applying an *edible coating as a carrier for anti-browning agent*. See, the Abstract, and page 3, lines 22-29. The acidulant used are *organic acids*, not mineral acids. See, page 5, lines 15-21. Applicant's invention *does not use* an edible coating as the carrier for an anti-browning agent.

It is respectfully submitted that the cited references, either singly or in combination, cannot teach or suggest Applicant's invention.